

UNIVERSITY OF TORONTO



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# PRACTICAL CHEMISTRY.

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HENRY CROFT, F.C.S.

J. Davis Barnett.  
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COURSE  
OF  
PRACTICAL CHEMISTRY

AS ADOPTED AT  
UNIVERSITY COLLEGE, TORONTO.

BY  
HENRY CROFT, F.C.S.  
PROFESSOR OF CHEMISTRY, UNIVERSITY COLLEGE.

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## P R E F A C E .

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In the present edition, only those tests have been introduced which, in the writer's experience, are likely to succeed, even in inexperienced hands. Many tests are apt to fail, such as some for manganese and chromium, &c. These have therefore been omitted ; but perhaps several good reactions may have been overlooked. The rarer elements have been disregarded, and only those referred to which are likely to come under the notice of a Canadian student. Uranium and Tellurium—rare substances in our country—have been omitted, as their ores would not probably come under the notice of any but a mineralogist.

The present edition having been entirely re-written under great press of work, many errors may doubtless have crept in.

H. H. CROFT, F.C.S.





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## NOTES ON TESTING.

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The test tubes used should be seven inches in length and three-quarters of an inch in diameter, so that they can be fully closed with the thumb. When so closed, and held between the first and second fingers, the liquid can be violently agitated, which often promotes the formation of a precipitate. If the solution be acid or hot, the tube may be held between the thumb and first two fingers, and agitated by the others. When it is necessary to boil or apply heat, it is always well to turn the mouth of the tube away from you to avoid accidents to yourself or fellow-workers. In examining a salt, one-half, about ten or twelve grains, should be dissolved in half a test tube full of distilled water by the aid of heat; and in the case of bismuth and a few other metals, by the addition of a few drops of nitric or hydrochloric acid—preferably nitric. For rendering insoluble substances soluble, see under the proper head. Beginners should commence with bodies that dissolve easily.

The solution so obtained is called the original solution, and should always be placed in one particular hole of the test tube stand, say the upper one on the left.

A portion of this, say half an inch, being poured out into a second tube, and diluted with about an equal bulk of water, the tests may be applied as in Table I. In adding hydrochloric acid, only a few drops should be used at first, and if a precipitate is formed, a little more added to see whether it dissolves or not. In using hydro-sulphuric acid, a large quantity should be employed, say an equal bulk; and it must be remembered that this reagent spoils very rapidly, and must often be prepared afresh. Some other reagents,

such as ammonium chloride, and calcium sulphate, must also be used in rather large quantity; the same with sodium phosphate. In using Table VII., the original solution should not be diluted.

In trying the solubility of the sulphides in ammonium sulphide or acid, it is better to allow the precipitate to settle, and to pour off as much of the liquid as possible; and the same applies to many other precipitates. In using ammonium sulphide, the solution will almost always become more or less yellow, and any light coloured precipitate the same, especially if the sulphide is not fresh. Very old sulphide is often quite colourless, and then, of course, useless.

Precipitates when formed rapidly from concentrated solutions are generally amorphous, without any visible crystalline structure, even when viewed under the magnifier; when formed slowly, and especially from dilute solutions, they are often highly granular and crystalline, sometimes forming on the sides of the tube. Much information may be derived by an examination of these crystals, either by the pocket lens or by a more powerful microscope.

The greatest care should be taken that the test tubes are perfectly clean. A piece of sponge, fastened on to a rod of wood eight or nine inches long, forms a convenient cleaner; the tubes being afterwards well rinsed with water. In most cases the latter only is required.

For ordinary working, the only requirements, in addition to tubes and stand, are—spirit lamp or gas burner, blowpipe, charcoal, platinum foil and wire, platinum crucible desirable, but a thin porcelain one will answer for many purposes; a few capsules or small dishes, funnels, filtering paper, glass rods and tubes, a pocket lens, and a few precipitating glasses, for larger quantities than could be contained in a test tube.

In using Table I., if no precipitate has been formed by hydrochloric acid, add hydrosulphuric acid to same solution, in small



quantity at first (test for mercuric salts), and at last an equal bulk ; if this produces nothing, or only a milkiness, add ammonium chloride in considerable quantity, then ammonia, until the liquid smells strongly after shaking ; and then, whether any change has taken place or not, a small quantity of ammonium sulphide. If no change is hereby produced, add sodium carbonate to the same solution, or perhaps preferably to a fresh portion of the original.

It may of course happen that in the examination of an unknown substance no base is found, the body being an acid, and *vice versa*. Having determined the nature of the base or acid by the tables, refer back to special tests under the respective heads, and go through all the reactions with small portions of the original solution, omitting in most cases those by which the groups have been determined.

SYMBOLS AND ATOMIC WEIGHTS OF THE COMMONER  
ELEMENTS.

Oxygen .....	O	16	Aluminum .....	Al	27.4
Sulphur .....	S	32	Manganese .....	Mn	55
Chlorine .....	Cl	35.5	Iron .....	Fe	56
Bromine .....	Br	80	Cobalt.....	Co	59
Iodine .....	I	127	Nickel .....	Ni	59
Fluorine .....	F	19	Chromium .....	Cr	52.4
Nitrogen .....	N	14	Zinc.....	Zn	65
Phosphorus.....	P	31	Cadmium .....	Cd	112
Carbon.....	C	12	Copper .....	Cu	63.4
Boron .....	B	11	Lead .....	Pb	207
Silicon .....	Si	28	Tin .....	Sn	118
Hydrogen .....	H	1	Bismuth .....	Bi	210
Potassium .....	K	39	Antimony .....	Sb	122
Sodium .....	Na	23	Arsenic .....	As	75
Lithium .....	L	7	Silver .....	Ag	108
Barium .....	Ba	137	Mercury.....	Hg	200
Strontium .....	Sr	87.5	Gold .....	Au	197
Calcium .....	Ca	40	Platinum .....	Pt	197.4
Magnesium.....	Mg	24			

## TABLE OF REAGENTS.

The formulas of these substances are given without reference to water of crystallisation, as they are generally used, except in preliminary or blowpipe tests, in form of solution. Where not mentioned, this will always be understood.

Sulphuric Acid .	Hydrogen Sulphate	$\text{H}^2\text{SO}^4$	$\text{SO}^2$ $\text{H}^2$	} $\text{O}^2$
Nitric Acid	Hydrogen Nitrate	$\text{HNO}^3$	$\text{NO}^2$ $\text{H}$	
Hydrochloric Acid	Hydrogen Chloride	$\text{HCl}$	$\text{H}$ $\text{Cl}$	} $\text{O}$
Hydrosulphuric Acid	Hydrogen Sulphide	$\text{H}^2\text{S}$	$\text{H}$ $\text{H}$	
Water	Hydrogen Oxide	$\text{H}^2\text{O}$	$\text{H}$ $\text{H}$	} $\text{O}$
* Oxalic Acid	Hydrogen Oxalate	$\text{H}^2\text{C}^2\text{O}^4$	$\text{C}^2\text{O}^2$ $\text{H}^2$	
Acetic Acid	Hydrogen Acetate	$\text{C}^2\text{H}^3\text{O}^2$	$\text{C}^2\text{H}^3\text{O}$ $\text{H}$	} $\text{O}$
Tartaric Acid	Hydrogen Tartrate	$\text{C}^4\text{H}^6\text{O}^6$	$\text{C}^4\text{H}^4\text{O}^4$ $\text{H}^2$	
Ammonia	Ammonium Hydrate, $\text{NH}^3\text{H}^2\text{O}$		$\text{NH}^4$ $\text{H}$	} $\text{O}$
Potassa	Potassium Hydrate	$\text{HKO}$	$\text{K}$ $\text{H}$	
Soda	Sodium Hydrate	$\text{HNaO}$	$\text{Na}$ $\text{H}$	} $\text{O}$
Slaked Lime	Calcium Hydrate	$\text{H}^2\text{CaO}^2$	$\text{Ca}$ $\text{H}^2$	
Sodium Carbonate		$\text{Na}^2\text{CO}^3$	$\text{CO}$ $\text{Na}^2$	} $\text{O}^2$
† Ammonium Carbonate..		$\text{Am}^2\text{CO}^3$	$\text{CO}$ $\text{Am}^2$	
Barium Chloride		$\text{BaCl}^2$	$\text{Ba}$ $\text{Cl}^2$	} $\text{O}^2$
Calcium Chloride		$\text{CaCl}^2$	$\text{Ca}$ $\text{Cl}^2$	
Magnesium Chloride, or Sulphate	}	$\text{MgCl}^2$	$\text{Mg}$ $\text{Cl}^2$	} $\text{O}^2$
		$\text{MgSO}^4$	$\text{SO}^2$ $\text{Mg}$	
Ammonium Chloride		$\text{AmCl}$	$\text{Am}$ $\text{Cl}$	} $\text{O}^2$
Ferric Chloride		$\text{Fe}^2\text{Cl}^6$	$\text{Fe}^2$ $\text{Cl}^6$	

\* Oxalic, Tartaric, Acetic and other Organic Acids, are often represented thus:  $\text{Ox}$ ,  $\text{T}$ ,  $\text{Ac}$ ,  $\text{Cl}$ ,  $\text{Tn}$ ,  $\text{Gl}$ , &c.

† The commercial salt is generally a sesquicarbonate, or contains some bicarbonate; hence ammonia is added.

Platinic Chloride.....	PtCl <sup>4</sup>	Pt Cl <sup>4</sup>	}
Stannous Chloride .....	SnCl <sup>2</sup>	Sn Cl <sup>2</sup>	
Copper Sulphate .....	CuSO <sup>4</sup>	SO <sup>2</sup> Cu	O <sup>2</sup>
Potassium Ferrocyanide....	K <sup>4</sup> Cfy	K <sup>4</sup> Cfy	}
Potassium Ferricyanide....	K <sup>3</sup> Cfy	K <sup>3</sup> Cfy	
Potassium Sulphocyanide ..	KCyS	K Cy	S
Potassium Chromate .....	K <sup>2</sup> CrO <sup>4</sup>	CrO <sup>2</sup> K <sup>2</sup>	O <sup>2</sup>
Sodium Phosphate .....	Na <sup>2</sup> HPO <sup>4</sup>	PO Na <sup>2</sup> H	O <sup>3</sup>
Sodium Acetate .....	NaC <sup>2</sup> H <sup>3</sup> O <sup>2</sup>	C <sup>2</sup> H <sup>3</sup> O Na <sup>2</sup>	O
Sodium Nitroprusside .....	Na <sup>2</sup> FeCy <sup>5</sup> NO (?)	Fe Cy <sup>5</sup> X Na	?
Ammonium Oxalate .....	Am <sup>2</sup> C <sup>2</sup> O <sup>4</sup>	C <sup>2</sup> O <sup>2</sup> Am <sup>2</sup>	O <sup>2</sup>
Ammonium Sulphide .....	Am <sup>2</sup> S	Am <sup>2</sup> S	}
Calcium Sulphate .....	CaSO <sup>4</sup>	SO <sup>2</sup> Ca	
Silver Nitrate .....	AgNO <sup>3</sup>	NO <sup>2</sup> Ag	O
Ammonium Molybdate ....	Am <sup>2</sup> MoO <sup>4</sup>	MoO <sup>2</sup> Am <sup>2</sup>	O <sup>2</sup>
Lead Acetate .....	Pb2C <sup>2</sup> H <sup>3</sup> O <sup>2</sup>	2C <sup>2</sup> H <sup>3</sup> O Pb	O <sup>2</sup>
Potassium Iodide .....	KI	I K	}
Sulphindigotic Acid .....	Ind		
Borax.....	Na <sup>2</sup> B <sup>3</sup> O <sup>4</sup>	} In blow- pipe tests.	
Microcosmic Salt.....	NaHAmPO <sup>4</sup>		

# ABBREVIATIONS USED IN FOLLOWING PAGES.

Precipitate .....	ppte.	Insoluble .....	insol.
Precipitant .....	pptt.	Volatile .....	vol.
Precipitated.....	pptd.	Sublimate.....	sub.
Precipitation .....	pptn.	Addition .....	adtn.
Solution .....	soln.	Original .....	orig.
Soluble .....	sol.		

TABLE I.—GROUP TESTS.

I. Adtn. of HCl to orig. soln. causes white ppt. insol. in excess of HCl.	II. Adtn. of H <sub>2</sub> S to same soln. causes ppt., the colour of which is, if H <sub>2</sub> S be added in excess,	III. Adtn. of Am <sup>2</sup> S to same soln. after adding excess of AmCl and NH <sub>3</sub> produces ppt., colour of which is	IV. Adtn. of Na <sub>2</sub> CO <sub>3</sub> to orig. soln. causes a white ppt., even in cold	V. No ppt. in orig. soln. by either of preceding re-agents.
Lead Silver Mercury in salts of lower oxide.	<i>Black.</i> Platinum Gold Lead Copper Mercury	<i>Black.</i> Iron Nickel Cobalt	Barium Strontium Calcium Magnesium	Potassium Sodium Ammonium Lithium
Antimony in organic salts may give a ppt. sol. in excess.	<i>Brown-Black.</i> Bismuth	<i>Buff.</i> Manganese		The last may give slight ppt. under IV. in concentrated solns. and heating.
The same with Tungstates and, to some extent, with Molybdates.*	<i>Brown.</i> Tin-stannous	<i>Greenish Gray.</i> Chromium		
Some chlorides, such as BaCl <sub>2</sub> , NaCl, &c., are pptd. by HCl, ppt. sol. in excess of water.	<i>Orange.</i> Antimony <i>Yellow.</i> Arsenic Tin-stannic Cadmium <i>White.</i>	<i>White.</i> Zinc Aluminum  These lighter colours may be much interfered with by presence of impurities. (See Table V.)		
	A ppt. or milkiness from S, in presence of salts of Fe <sup>2</sup> O <sub>3</sub> , CrO <sub>3</sub> , Cl, &c.	If from acid solns., phosphates or oxalates may be present.		

\* See Table II.



TABLE II.—GROUP I.\*

Adtn. of $\text{NH}_3$	Adtn. of $\text{NH}_3$	Adtn. of $\text{NH}_3$	The adtn. of a piece of Zn. to the acid soln.
Scarcely changes the ppte. Orig. soln. gives with $\text{H}^2\text{SO}^4$ a white ppte. The ppte. by $\text{HCl}$ is sol. in much boiling water.	Disolves the ppte. Orig. soln. gives a gray ppte. with $\text{FeSO}^4$ . White ppte. turns gray or black on exposure to sunlight.	Turns the ppte. gray or black. Orig. soln. gives with $\text{H}^2\text{SO}^4$ a white ppte. On polished copper or gold a gray stain removed on heating.	Gives dark blue colour after a short time. The acid soln., heated with a drop of sodium phosphate, gives a yellow ppte. Molybdic acid.
Orig. soln. gives yellow ppte. with $\text{K}^2\text{CrO}^4$ . Lead.	Orig. soln. gives with $\text{K}^2\text{CrO}^4$ a scarlet ppte. Silver.	The white ppte., when boiled with $\text{HCl}$ , turns gray. Mercurous salts.	Produces no yellow ppte. Tungstic or Metatungstic acid. See page 19. For antimony, see Table III.

\* The ppte. formed on addition of  $\text{HCl}$  should be allowed to settle, the liquid poured off, and fresh portions of water added, the operation repeated. The settlement will generally be aided by violent agitation. This supposing the ppte. is insol. in moderate excess of  $\text{HCl}$ .

TABLE III.—GROUP II (a).

Ppte. sol. in  $\text{Am}^2\text{S}$ , alone or on actn. of S, Ppte. is

Black.	Black.	Yellow.	Yellow.	Orange.
Orig. soln. gives with $\text{KCl}$ a yellow, more or less cryst. ppte.	Orig. soln. gives with $\text{FeSO}_4$ a blue colour or brown ppte.	Washed ppte. is sol. in $\text{NH}_3$ .	Washed ppte. is insol. in $\text{NH}_3$ . Orig. soln. gives with $\text{HKO}$ a white ppte. sol. in excess.	Washed ppte. is sol. in $\text{HKO}$ .
With $\text{KI}$ a brown colour or dark ppte.	With $\text{Ox}$ on warming a blue colour, and ppte. of brown metal, sometimes on the glass with golden lustre.	Orig. soln. gives with $\text{HKO}$ no ppte.	Dry salt heated on charcoal with $\text{Na}^2\text{CO}_3$ and $\text{KCy}$ gives metallic beads sol. in hot $\text{HCl}$ , the soln. showing reactions of tin. The mass well fused may be picked out, ground with a little water in agate mortar, liquid poured off, when metallic scales will appear; same with copper, &c.	Orig. soln. containing $\text{HCl}$ gives with zinc a black ppte. and gas burning with white flame.
		Dry salt heated in tube with dry $\text{Na}^2\text{CO}_3$ and $\text{KCy}$ evolves garlick odour, and forms ring of brown colour or metallic lustre.	If the orig. ppte. formed by $\text{H}^2\text{S}$ was brown, stannous salt would be indicated. If yellow, then stannic salt.	Acid soln. gives on platinum foil when touched with zinc a black stain.
Platinum.	Gold.	Arsenic.	Tin.	Antimony.

TABLE IV.—GROUP II (b).

Ppte. insol. in fresh  $\text{Am}^2\text{S}$ , and even after adtn. of  $\text{S}$ , or use of old  $\text{Am}^2\text{S}$ , ppte. is

Black.	Black with excess of $\text{H}^2\text{S}$ .	Brown-Black.	Brown-Black.	Yellow.
Orig. soln. gives with $\text{H}^2\text{SO}^4$ a white ppte. With $\text{HCl}$ a white ppte. sol. in excess of boiling water. With $\text{KI}$ a yellow ppte. sol. in large excess of boiling water.	Orig. soln. gives bright yellow with $\text{HKO}$ in excess. White turning to gray with $\text{SnCl}^2$ , especially on heating. Stain on copper.	Orig. soln. gives with $\text{HKO}$ a blue ppte., turning black on heating with excess. Orig. soln. gives a fine blue colour with excess of $\text{NH}^3$ .	Orig. salt does not dissolve in water, even on boiling, without aid of $\text{HCl}$ or $\text{HNO}^3$ . Gives ppte. with $\text{HKO}$ , insol. in excess. Gives with $\text{K}^2\text{CrO}^4$ yellow ppte. insol. in excess.	Orig. soln. with $\text{NH}^3$ gives ppte. sol. in excess. Orig. soln. with $\text{HKO}$ gives ppte. insol. in excess.
Lead.	Mercury.	Copper.	Bismuth.	Cadmium.

\* In making this experiment, it is well to throw away the larger portion of the ppte., and act only on a small quantity. The same applies to all similar experiments.

TABLE V.—GROUP III.

DIVISION I.—PTE. IS BLACK.

The black sulphide should be allowed to settle, and washed several times with water, especially if nitric or chloric acid be present.

Disolves it.		The addition of hydrochloric acid to the washed sulphide	
Original solution gives with potassium hydrate or ammonia a dirty green precipitate, turning black and then brown.	Original solution gives with potassium hydrate or ammonia a brown precipitate. Organic acids interfere with these reactions.	Original solution gives with potassium hydrate a green precipitate, soluble in ammonia with a blue colour.	Original solution gives with potassium hydrate a blue precipitate, soluble in ammonia with a brown colour.
Iron in ferrous salts.	Iron in ferric salts.	Nickel.	Cobalt.

DIVISION II.—PTE. IS NOT BLACK.

The precipitates are all soluble in hydrochloric acid, and their colours are Greenish, or Gray.

Buff. Original solution gives with potassium hydrate a white precipitate, turning brown on exposure to the air.	Original solution gives with potassium hydrate a green or gray precipitate, soluble in excess to a fine green solution.	Original solution gives with ammonia a white precipitate, soluble in excess. The same with potassium hydrate.	White. Original solution gives with ammonia a white transparent precipitate, insoluble in excess. The same with potassium hydrate, but soluble in excess.
Manganese.	Chromium.	Zinc.	Aluminum.

This colour is often not pure buff, but grayish, if the salt be not pure. The same is the case with the precipitate formed from zinc and aluminum solutions. The tests with potassium hydrate or ammonia will decide the point.

From acid solutions white precipitates may be formed, consisting of earthy phosphates, oxalates, &c.



TABLE VI.—GROUP IV.

If the solution employed in the previous group tests is employed, that is, if salts of ammonia are present, it may be better to warm gently. With original solution this is not necessary.

The original solution, mixed with a large quantity of ammonium chloride and a moderate quantity of ammonium carbonate, gives

A white precipitate.			No precipitate.
Original solution gives, with calcium sulphate,			The addition of sodium phosphate produces a precipitate, crystalline when from dilute solutions.
A precipitate at once.	A precipitate after some time.	No precipitate.	
The salt colours the flame of alcohol green.	The salt colours the flame of alcohol crimson.	The salt colours the flame of alcohol orange red.	
Barium.	Strontium.	Calcium.	Magnesium.

TABLE VII.—GROUP V.

Original solution, rather concentrated, and acidulated with hydrochloric acid, gives, with platinum tetrachloride,

A yellow precipitate.		No precipitate.	
Dry salt heated with potassium hydrate, evolves no ammonia.	Dry salt heated with potassium hydrate, evolves ammonia.	Salts colour the flame of alcohol yellow.	Salts heated in blow-pipe flame produce a magnificent purple colour.
Dry salt colours the flame of alcohol violet.	Salts are volatile, and do not colour the flame of alcohol.		
Potassium.	Ammonium.	Sodium.	Lithium.

If no metal is found, the substance is probably an acid, and the base hydrogen.



## NOTES ON TESTING FOR ACIDS.

If in solution, and the blackening effect of heat is to be tried, HKO may be added, and the whole evaporated to dryness. Salts should be tested as to their neutrality; if acid, a little ammonia may be added till alkaline and warmed. Many salts of the heavy metals cannot thus be neutralized, as the least trace of ammonia produces a precipitate, such salts being inherently acid.

It is always well to detect the base or metal first; this is specially the case with ammonium, some organic salts of which do not blacken when heated, and give no solid, but only a liquid sublimate. Moreover, many metals form insoluble salts with several acids; hence if the salt is easily soluble in water, these need not be looked for.

Ba, in soluble salt, precludes  $\text{H}^2\text{SO}^4$ ,  $\text{H}^2\text{CO}^3$ ,  $\text{H}^3\text{PO}^4$ , &c.

Pb precludes  $\text{H}^2\text{SO}^4$ , Cl (?) Br, I,  $\text{H}^2\text{CO}^3$ ,  $\text{H}^3\text{PO}^4$ , &c.

Ag precludes Cl, Br, I, and so on.

TABLE VIII.—GROUPS

I. Salts, excepting some of $\text{NH}_4^+$ , blackening more or less when heated in a close tube.	II. Salts not blackening when heated giving a ppte. with $\text{BaCl}_2$ in neutral solutions.	III. Salts not blackening giving no ppte. with $\text{BaCl}_2$ , but with $\text{AgNO}_3$ in neutral or acid solutions.	IV. Salts deflagrating when heated on charcoal.
<p><i>Strongly</i></p> <p>Tartaric Citric Tannic Gallic Uric Benzoic Acetic</p> <p><i>Slightly</i></p> <p>Oxalic Ferrocyanic Ferrycyanic Sulphocyanic</p> <p>Many salts of ammonia volatilise or are decomposed without blackening.</p>	<p>Sulphurous Sulphuric Phosphoric Boracic Silicic Carbonic Hydrofluoric Oxalic (1st Group) Ferrocyanic (1st Group) Chromic Arsenic Arsenious</p>	<p>Hydrochloric Hydrobromic Hydriodic Hydrocyanic Hydrosulphuric Ferrycyanic } 1st Sulphocyanic } Group.</p>	<p>Nitric Chloric Iodic Bromic Perchloric</p> <p>Lead oxide also deflagrates to some extent when heated on charcoal. Detected at once by formation of globules of metallic lead. <math>\text{AmNO}_3</math> is decomposed with slight deflagration.</p>

TABLE IX.—GROUP I.

Salts blackening in tube more or less.

I.	II.
<p>Salts when heated evolving smell of burnt sugar, blackening rapidly when heated with <math>\text{H}^2\text{SO}^4</math>. Acid or salt with very large excess of lime water, gives white ppte., sol. in very large excess of <math>\text{HKO}</math>; give dark colour when boiled with <math>\text{AgNO}^3</math>. Acid gives ppte. with little <math>\text{HKO}</math> on shaking.</p>	<p>Salts blacken very slightly, residue dissolves in acids with effervescence. Dry salt or acid heated with <math>\text{H}^2\text{SO}^4</math> evolves a gas burning with blue flame. Ppte. with <math>\text{CaCl}^2</math> insol. in acetic acid. With <math>\text{MnO}^2</math> and <math>\text{H}^2\text{SO}^4</math> violent effervescence.</p>
Tartaric.*	Oxalic.
<p>Evolving acid smell when heated, blackening slowly with hot <math>\text{H}^2\text{SO}^4</math>. On boiling, white ppte., with excess of lime water, soluble in <math>\text{NH}^3</math>. No ppte. when shaken with little <math>\text{HKO}</math>.</p>	<p>Salts turn brown on heating; boiled with dilute <math>\text{H}^2\text{SO}^4</math> give off smell of prussic acid, or with excess of strong <math>\text{H}^2\text{SO}^4</math>, a gas burning with blue flame <math>\text{CO}</math>.</p>
Citric.	Ferrocyanic.
<p>Blackens rapidly with <math>\text{H}^2\text{SO}^4</math>, gives blue-black colour with <math>\text{Fe}^2\text{Cl}^6</math>, precipitates gelatine.</p>	<p>Salts turn brown on heating. Give dark blue colour with ferrous salts, brown colour with ferric.</p>
Tannic.	Ferrieyanic.
<p>Blackens rapidly with <math>\text{H}^2\text{SO}^4</math>, blue-black colour with <math>\text{Fe}^2\text{Cl}^6</math>, does not ppte. gelatine.</p>	<p>Salts darken on heating. Dry mercuric salt swells up to voluminous ash. Red colour with <math>\text{Fe}^2\text{Cl}^6</math>, destroyed by <math>\text{HgCl}^2</math>, not by boiling. (See page 19.)</p>
Gallic.	Sulphocyanic.
<p>Dry salt heated with <math>\text{H}^2\text{SO}^4</math> evolves smell of vinegar, with alcohol added the sweet smell of acetic ether; with <math>\text{Fe}^2\text{Cl}^6</math>, red colour.</p>	
Acetic.	
<p>Heated with <math>\text{H}^2\text{SO}^4</math>, evolves pungent vapour. Solution gives buff ppte. with <math>\text{Fe}^2\text{Cl}^6</math>.</p>	
Benzoic.	
<p>Acid or salt, evaporated to dryness in capsule, with few drops of <math>\text{HNO}^3</math> gives red colour, turning purple with <math>\text{HKO}</math>.</p>	
Uric.	

\* In ammoniacal solutions of tartrates,  $\text{CaCl}^2$  produces a flocculent ppte, which soon becomes crystalline and insol. in acetic acid. It might thus be mistaken for oxalate, but this latter is dissolved by  $\text{HCl}$ , and reprecipitated by  $\text{NH}^3$ , while the tartrate is not reprecipitated. The strong blackening of the latter is also characteristic.

TABLE X.—GROUP II.

I. Salts treated with moderately strong $\text{H}_2\text{SO}_4$ , evolve a gas.	II. Salts do not evolve a gas with $\text{H}_2\text{SO}_4$ . Give a ppte. with $\text{BaCl}_2$ .
<p>Without smell.</p> <p>The gas decanted into a tube, and shaken with lime water gives a white ppte.</p> <p>Carbonic</p> <p>Smells of burning sulphur.</p> <p>Gas acts on Iodic Starch paper, producing blue colour.</p> <p>Salts boiled with Zn and HCl evolve <math>\text{H}_2\text{S}</math>, which blackens lead paper.</p> <p>Sulphurous</p> <p>Sol. salts evolve with acids <math>\text{H}_2\text{S}</math>, recognised by smell, and as above</p>	<p>Insol. in dilute <math>\text{HNO}_3</math>.</p> <p>Salts heated on charcoal with <math>\text{Na}_2\text{CO}_3</math> give a red mass, which when moistened forms a brown stain on silver.</p> <p>The red mass evolves <math>\text{H}_2\text{S}</math> when treated with dilute <math>\text{H}_2\text{SO}_4</math>, and if dissolved in water gives fine purple colour with sodium nitroprusside.</p>
<p>Sol. salts evolve with acids <math>\text{H}_2\text{S}</math>, recognised by smell, and as above</p> <p>Hydrosulphuric.</p>	<p>Sulphuric</p>
<p>Sol. in dilute <math>\text{HNO}_3</math>.</p> <p>Salts give a yellow ppte. with <math>\text{AgNO}_3</math>, and yellow with ammonium molybdate, especially on warming.</p> <p>Phosphoric</p> <p>Salts, treated with <math>\text{H}_2\text{SO}_4</math> and burnt with alcohol, give green flame. Also free acid in blow-pipe flame. See Blow-pipe Tests.</p> <p>Acid, when set free by HCl, turns turmeric paper dark brown red.</p> <p>Boric</p> <p>Acid separated from its strong solutions as a jelly by HCl. On evaporating to dryness and treating residue with <math>\text{H}_2\text{O}</math>, becomes more visible and not gelatinous.</p> <p>Silicic</p> <p>Salts are red, yellow or brown. Yellow ppte. with lead acetate, scarlet with <math>\text{AgNO}_3</math>, turned green by boiling with HCl and alcohol or sulphurous acid.</p> <p>Chromic</p> <p>Brown red ppte. with <math>\text{AgNO}_3</math>, white ppte. with <math>\text{MgSO}_4</math> and <math>\text{NH}_3</math>.</p> <p>Arsenic.</p>	<p>(For Oxalic, Hydrofluoric and Ferricyanic, see Tables IX., XI., and page 19.)</p>



TABLE XI.—GROUP III.  
Ppte. with  $\text{AgNO}_3$  is insol. in dilute  $\text{HNO}_3$ .

<i>Black.</i>	<i>Yellowish.</i>	<i>Brown- Yellow.</i>	<i>White.</i>
<p>Sol. salts give a purple colour with sodium nitroprus- side.</p> <p>Black ppte. with salts of lead, many evolve <math>\text{H}_2\text{S}</math> with acids.</p> <p>Dry salts heated with <math>\text{HNO}_3</math>, evolve red fumes, and the soln. contains <math>\text{H}_2\text{SO}_4</math>.</p> <p>Hydrosulphuric or sulphur.</p>	<p>Insol. in <math>\text{NH}_3</math>.</p> <p>Dry salt heated with <math>\text{H}_2\text{SO}_4</math> evolves violet fumes.</p> <p>Solutions treated with <math>\text{Cl}</math> become yellow or brown, giv- ing a blue colour with starch and a pink or dark colour when shaken with carbon disulphide, benzol or chloro- form.</p> <p>Hydriodic or iodine.</p> <p>Sol. in much hot <math>\text{NH}_3</math>.</p> <p>Dry salts heated with <math>\text{H}_2\text{SO}_4</math> evolve white and yellow fumes.</p> <p>Solutions treated with <math>\text{Cl}</math> and shaken with ether, yield to it a yellow colour, and the aqueous liquid becomes colour- less.</p> <p>Hydrobromic or bromine.</p>	<p>Solutions give brown colour with <math>\text{Fe}^{+}\text{Cl}^6</math>.</p> <p>Ferricyanic.</p> <p><i>Dull White.</i></p> <p>Solutions give blue ppte. with <math>\text{Fe}^{+}\text{Cl}^6</math>.</p> <p>Ferrocyanic, salts of which acid give a ppte. with <math>\text{BaCl}_2</math>, insol. in <math>\text{Ac}</math>, hence resembling oxalates. For this reason <math>\text{H}^{+}\text{Cfy}</math> appears in two groups.</p> <p>Solutions give blood-red colour with <math>\text{Fe}^{+}\text{Cl}^6</math>.</p> <p>Sulphocyanic.</p>	<p>Salts heated with <math>\text{H}_2\text{SO}_4</math> evolve pungent fumes of <math>\text{HCl}</math>.</p> <p>On addition of <math>\text{MnO}_2</math> evolve <math>\text{Cl}</math>, recognized by smell and bleaching power.</p> <p>Hydrochloric or chlorine.</p> <p>Salts heated with <math>\text{H}_2\text{SO}_4</math> evolve <math>\text{HCl}</math>, recognized by odour, by being absorbed by <math>\text{Am}^+\text{S}</math>, and on evaporation giv- ing a red colour with <math>\text{Fe}^{+}\text{Cl}^6</math>.</p> <p>Solutions mixed with both ferrous and ferric salts, pptd. by <math>\text{HKO}</math> and again dissolved in <math>\text{HCl}</math>, give blue colour or ppte.</p> <p>Hydrocyanic or Cyanogen.</p>



TABLE XII.—GROUP IV.

No ppte. with $\text{BaCl}^2$ .  No ppte. with $\text{AgNO}^3$ till after heating the dry salt.  Salts treated with $\text{H}^2\text{SO}^4$ turn reddish and evolve a gas $\text{Cl}^2\text{O}^4$ which is very explosive.  On addn. of $\text{HCl}$ or $\text{H}^2\text{SO}^4$ destroys colour of $\overline{\text{Ind}}$ .	A ppte. with $\text{BaCl}^2$ .  When heated on charcoal gives off violet fumes of iodine.  Treated with $\text{SO}^3$ or salts in presence of $\text{HCl}$ and starch gives blue colour.  Iodate in the iodide detected by tartaric acid by brown colour produced, and action on starch.	A ppte. with $\text{BaCl}^2$ .  No ppte. with $\text{AgNO}^3$ till after dry salt has been strongly heated.  Salts with $\text{H}^2\text{SO}^4$ evolve bromine.  Bromate mixed with bromide detected by yellow colour produced on addition of tartaric acid. The bromine can be extracted from the liquid by ether.	No ppte. with $\text{BaCl}^2$ .  Treated with $\text{H}^2\text{SO}^4$ does not evolve explosive gas if free from chloric.  No ppte. with $\text{AgNO}^3$ till after the dry salt has been strongly heated.  Same action on $\overline{\text{Ind}}$ as chloric acid on addition of $\text{HCl}$ .  Scarcely any action if free from chloric when $\text{H}^2\text{SO}^4$ is used instead of $\text{HCl}$ .
No ppte. with $\text{BaCl}^2$ unless in very concentrated solns. ppte. sol. in much water.  Dry salt heated with $\text{H}^2\text{SO}^4$ and metallic copper evolves red fumes.  Salt or soln. heated with $\text{H}^2\text{SO}^4$ destroys the blue colour of $\overline{\text{Ind}}$ .			Nitric.
	Chloric.	Iodic.	Perchloric.

REACTIONS OF SOME ACIDS NOT MENTIONED IN  
PRECEDING TABLES.

Acetic acid or salts give a red colour with  $\text{Fe}^2\text{Cl}^6$ , destroyed by  $\text{HCl}$  and by boiling, with formation of a precipitate, red colour of soln. is not destroyed by  $\text{HgCl}^2$ .

Meconic acid or salts give a red colour with  $\text{Fe}^2\text{Cl}^6$ , not destroyed by  $\text{HCl}$ , by boiling or by  $\text{HgCl}^2$ .

Sulphocyanic acid or salts give a red colour with  $\text{Fe}^2\text{Cl}^6$ , not destroyed by  $\text{HCl}$  or by boiling, but by  $\text{HgCl}^2$ .

Salicylic acid or salts give a purple colour with  $\text{Fe}^2\text{Cl}^6$ . Heated with lime evolve the peculiar odour of phenol or carbolic acid.

Carbolic acid, recognized by odour, by giving a brown colour with alcoholic  $\text{Fe}^2\text{Cl}^6$ ,\* by giving when heated with  $\text{HNO}^3$ , carbazotic acid which forms yellow ppte. with salts of potassium. By giving whitish ppte. with bromine water.

Hydrofluoric or salts give a ppte. with  $\text{CaCl}^2$ , and with  $\text{BaCl}^2$ , which is often not visible at first. No ppte. with  $\text{AgNO}^3$ . Salts of fluorine best detected by heating with  $\text{H}^2\text{SO}^4$ , when a gas is evolved, which acts on glass.

Tungstic acid in salts gives ppte. with  $\text{HCl}$  sol. in excess, giving blue colour with zinc, metatungstates give little or no ppte.

Molybdic acid in salts gives ppte. very easily sol. in excess, dark blue with zinc, salt with  $\text{H}^2\text{S}$ , brownish colour and brown ppte. on addtn. of acid. For test by phosphoric acid, see Table II.

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\* Hereby distinguished from creosote, which gives dark green.

## SUPPLEMENTARY TABLE.

May be used with solutions, to avoid evaporation. Also in examining salts of ammonium, some of which volatilise or are decomposed without blackening. A fresh portion of the solution should be used for each column, and thus the acids reduced to few in number. Solutions may be made neutral by addition of  $\text{NH}_3$  and warming.

Pptd. from neutral solns. by $\text{BaCl}_2$ .	Pptd. from neutral solns. by $\text{CaCl}_2$ .	Pptd. from neutral solns. by $\text{AgNO}_3$ .	Neutral solns. give with $\text{FeCl}_3$ .	Dry salts deliquesce on charcoal.
Ppte. insol. in $\text{HNO}_3$ or $\text{HCl}$ .	Ppte. formed at once.	Ppte. insol. in dilute $\text{HNO}_3$ .	White Ppte. Phosphoric	Nitric Chloric Iodic Bromic Perchloric
Sulphuric	Insoluble in $\text{Ac}$ .	Black. Hydrosulphuric	Blue Ppte. Ferrocyanic	
Ppte. is soluble in $\text{HNO}_3$ or $\text{HCl}$ with effe- vescence.	Oxalic Hydrofluoric Ferrocyanic	White. Hydrochloric Hydrocyanic Ferrocyanic Sulphocyanic	Brown Colour. Ferrocyanic Hydriodic	
Carbonic Sulphurous* Without effervescence.	Soluble in $\text{Ac}$ .	Yellowish. Hydriodic Hydrobromic	Red Colour. Acetic Sulphocyanic	
Phosphoric Boric Oxalic Hydrofluoric Tartaric Citric Chromic Arsenious Arsenic	Carbonic Phosphoric Boric Tartaric Arsenious Arsenic	Brownish. Ferrocyanic		
	Ppte. formed after boiling, Citric	Ppte. sol. in dilute $\text{HNO}_3$ . White. Boracic Oxalic Tartaric Citric Yellow. Phosphoric Arsenious Red Brown. Chromic Arsenic		

\* The dry salt will exhibit effervescence, and also the ppte. if freed from water; but as  $\text{SO}_4$  is easily sol. in  $\text{H}_2\text{O}$ , other tests, such as smell, and action on iodic starch paper, had better be relied on.

# REAGENTS

REQUIRED IN THE USE OF PRECEDING TABLES, OR IN EXPERIMENTS  
HEREAFTER DESCRIBED.

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The acids  $\text{H}^2\text{SO}^4$ ,  $\text{HNO}^3$ ,  $\text{HCl}$  and  $\text{KHO}$ , should be kept in stoppered bottles. All glass containing lead should be discarded. Some solutions spoil on keeping, such as tartaric acid, ferricyanide, &c.; hence they must be prepared when required.

A little experience is necessary in the use of these reagents as to quantity required;  $\text{H}^2\text{S}$ ,  $\text{CaSO}^4$ ,  $\text{CaH}^2\text{O}^2$  should be used in large amounts, perhaps twice or thrice the bulk of the liquid under examination. Others, such as  $\text{AmCl}$ ,  $\text{Na}^2\text{HPO}^4$ , may be used in smaller quantities, while  $\text{Am}^3\text{S}$ ,  $\text{AgNO}^3$ ,  $\text{BaCl}^2$ , and others, may be employed in very small quantities.

In trying the solubility of a substance in any reagent, it is well to use only a small quantity of the body to be experimented on; such as  $\text{PbO}$ ,  $\text{PbCrO}^3$ , &c.

## REAGENTS USED IN THE DRY FORM.

*Borax*,  $\text{Na}^2\text{B}^4\text{O}^7 + 2\text{H}^2\text{O}$ , should be heated, when it swells up and loses all water of crystallisation, and then powdered. A loop being formed on a piece of platinum wire and heated red hot, will cause some of the powder to adhere when dipped into it. This can be fused in the outer blowpipe flame, and the operation repeated till a clear bead is produced.

*Microcosmic salt*, phosphor salt,  $\text{AmHNaPO}^4 + 4\text{H}^2\text{O}$ , should be heated till most of the water of crystallisation is given off. It can then be used for producing a bead either on charcoal or platinum wire. The bead consists of sodium metaphosphate, which, like borax, possesses the power of dissolving many oxides.  $\text{NH}^4\text{HNaPO}^4 = \text{NaPO}^3 + \text{NH}^3 + \text{H}^2\text{O}$ .

*Sodium carbonate*, obtained by heating to dull redness pure sodium bicarbonate,  $2\text{NaHCO}^3 = \text{Na}^2\text{CO}^3 + \text{CO}^2 + \text{H}^2\text{O}$ . For decomposing silicates, earthy sulphates and many other substances, a mixture of



sodium and potassium carbonates is preferable, as it fuses more readily than either. It may be made by mixing equal parts of the bicarbonates, these being always purer than the carbonates. Formula,  $\text{NaKCO}_3$ .

*Potassium nitrate*,  $\text{KNO}_3$ , common nitre purified by recrystallisation.

*Potassium bichromate*,  $\text{KCrO}_4\text{CrO}_3$ , purified as above.

*Potassium cyanide*,  $\text{KC}_y$ , pure enough as used by photographers.

*Calcium fluoride*,  $\text{CaF}_2$ , and *cupric oxide*,  $\text{CuO}$ , may be required.

*Potassium bisulphate*,  $\text{KHSO}_4$ , obtained by heating to fusion residue from preparation of  $\text{HNO}_3$ , or by adding one ounce of  $\text{HSO}_4$  to two ounces of official  $\text{KSO}_4$ , and evaporating to crystallisation.

## REAGENTS USED IN LIQUID FORM.

### SULPHURIC ACID—HYDRIC SULPHATE.

#### $\text{H}^2\text{SO}_4$ .

Commercial acid usually contains some lead, as  $\text{PbSO}_4$ . Its presence may best be detected by pouring some hydrochloric acid ( $\text{HCl}$ ), diluted with an equal bulk of water, on to some of the  $\text{H}^2\text{SO}_4$  contained in a test tube, taking care that the liquids do not mix. A white film at the line of junction will indicate lead, caused by  $\text{PbCl}_2$ . Pb. 814

$\text{H}^2\text{SO}_4$  sometimes, but not often, contains nitric acid,  $\text{HNO}_3$ ; this may be detected by pouring on to the acid, as described above, a solution of ferrous sulphate,  $\text{FeSO}_4$ . A brown film at the line of junction will indicate  $\text{HNO}_3$ , owing to formation of  $\text{N}^2\text{O}^2$ , which gives dark colour with  $\text{FeSO}_4$ . H<sup>2</sup>O<sub>3</sub>

$\text{H}^2\text{SO}_4$  often contains arsenic in form of arsenious acid,  $\text{As}^2\text{O}^3$ , derived from the iron pyrites  $\text{FeS}^2$ , often mixed with arsenical pyrites  $\text{FeSAs}$ , from which the sulphur used in the manufacture is obtained. It may be detected by diluting the acid with four or five times its bulk of water, and passing sulphuretted hydrogen (hydric sulphide  $\text{H}^2\text{S}$ ) through it. A yellow precipitate indicates arsenic. See further tests under arsenic. Sulphurous acid (sulphur dioxide,  $\text{SO}^2$ ) is sometimes present. For detection, see under sulphurous acid. Presence

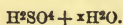
The  $\text{HNO}_3$  in  $\text{H}^2\text{SO}_4$  may also be detected by mixing the acid with an equal bulk of water, cooling, and throwing in a crystal of ferrous sulphate; if  $\text{HNO}_3$  be present, a brown colouration will appear round the crystal.



Also by heating the acid with a drop of the blue solution of indigo (sulphindigotic acid); if  $\text{HNO}^3$  be present, the blue colour will be destroyed and a yellow appear.

In this, as in all following cases, it will be well for the student, having a pure article under his hands, to add small quantities of the impurities mentioned, and to go through the required tests for the sake of practice.

#### DILUTE SULPHURIC ACID.



One part of the strong acid poured into six or eight measured parts of water; the acid should always be poured into the water with constant agitation, and not the reverse. As the heat evolved may cause a breakage of the glass, it is better to mix in a porcelain dish. With common acid the mixture is often turbid from separation of lead sulphate,  $\text{PbSO}^4$ ; in such case, allow to settle, pour off or filter.

#### SULPHUROUS ACID—HYDRIC SULPHITE.



By passing  $\text{SO}^2$  from Cu and  $2\text{H}^2\text{SO}^4$  into water to saturation. It spoils on exposure to the air, and changes by oxidation to  $\text{H}^2\text{SO}^4$ . The pure acid gives a precipitate with  $\text{BaCl}^2$ , soluble in slight excess of HCl. If  $\text{H}^2\text{SO}^4$  has been formed, the precipitate is partly insoluble.

A solution of sodium bisulphite  $\text{NaHSO}^3$  (hydric sodium sulphite) is sometimes used instead of the acid, being more permanent. Obtained by passing  $\text{SO}^2$  into solution of sodium carbonate to full saturation. Oxidation detected as above. This salt or its solution is now commercial.

In making  $\text{SO}^2$  for these purposes, always use a safety tube attached to the flask or retort; the running back of the water or soda solution, may cause a dangerous explosion.

#### NITRIC ACID—HYDRIC NITRATE.



Sometimes contains hydrochloric, detected by silver nitrate,  $\text{AgNO}^3$ , added to diluted acid; a white curdy precipitate,  $\text{AgCl}$ , indicates the presence of chlorine. To purify, distil until the product produces no precipitate with  $\text{AgNO}^3$ , the residue is free from Cl. Sometimes contains sulphuric acid, detected by adding barium chloride to the largely diluted acid. A white precipitate of barium sulphate,  $\text{BaSO}^4$ , H<sub>2</sub>SO<sub>4</sub>

indicates the presence of  $\text{H}^2\text{SO}^4$ . To purify, add a small quantity of barium nitrate,  $\text{Ba}^2\text{NO}^3$ , allow to settle, pour off and distil. Commercial acid is generally nearly pure.

The strongest nitric acid is obtained by distilling equal parts by weight of pure nitre,  $\text{KNO}^3$ , and  $\text{H}^2\text{SO}^4$ ; this is required in some cases, but is not absolutely necessary.

#### HYDROCHLORIC ACID—HYDRIC CHLORIDE.



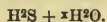
The commercial acid often contains arsenic, detected as already described, and also by more accurate tests mentioned under Arsenic. It not unfrequently contains iron, detected by neutralising the diluted acid with ammonia,  $\text{NH}^3$ , and adding a few drops of ammonium sulphide ( $\text{Am}^2\text{S}$ ). A black precipitate or green colouration indicates the presence of iron.  $\text{HCl}$  sometimes contains free chlorine, detected by its bleaching or decolourising action on solution of indigo (sulphin-digotic acid) when warmed.

To purify, dilute to one half, pass sulphuretted hydrogen through for some hours, warm gently and filter. This separates the arsenic but not the iron.

Sulphuric acid may be detected in the *largely* diluted acid by means of barium chloride, but cannot be separated by above process; sulphurous acid by blueing action on iodic-starch paper, or by allowing the acid to act on zinc, and exposing to the evolved gas a piece of paper moistened or smeared with lead salt. A darkening shews presence of  $\text{SO}^2$  from formation of  $\text{H}^2\text{S}$ ;  $\text{SO}^2 + \text{H}^6\text{Cl}^6 + \text{Zn}^3 = \text{H}^2\text{S} + \text{H}^4\text{O}^2 + \text{Zn}^3\text{Cl}^6$ .

Pure hydrochloric acid can generally be obtained. The above remarks apply only to the crude article.

#### HYDROSULPHURIC ACID—HYDRIC SULPHIDE— SULPHURETTED HYDROGEN.

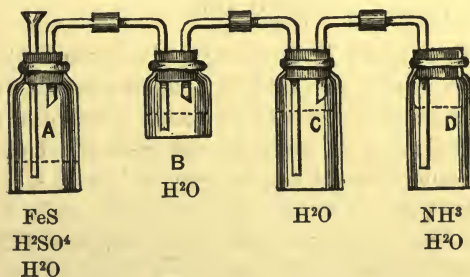


This reagent is used either as a gas or as solution in water, which rapidly deteriorates and must be constantly renewed. The want of the well known and disagreeable smell of  $\text{H}^2\text{S}$  will at once shew that the solution is useless.

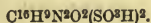
It should always be used in large quantity, unless perhaps in testing for mercuric salts, when the gradual addition may lead at once to the required result.

$\text{H}^2\text{S}$  is made from iron sulphide and dilute sulphuric acid.  $\text{FeS} + \text{H}^2\text{SO}^4$ , with four parts of water,  $= \text{H}^2\text{S} + \text{FeSO}^4$ .  $\text{FeS}$  may be made by heating together thirty parts of iron filings or turnings, and twenty parts of sulphur. It is best to throw small portions of this mixture into a red-hot crucible, waiting after each addition until the excess of sulphur is burnt off.

In making the solution in water, a large portion of the gas is wasted; it may be utilized by arranging the apparatus as below. The gas is washed in the first small bottle, then passes into the larger one for making the required solution, and then into a bottle containing dilute ammonia. Thus the ammonium sulphide may be prepared at the same time.  $2\text{NH}^3 + \text{H}^2\text{S} = (\text{NH}^4)^2\text{S}$ . The solution is usually  $(\text{NH}^4)^2\text{S} + \text{H}^2\text{S}$ . To make this perfectly pure, without any excess of  $\text{NH}^3$ , the gas should be passed in until the reagent produces no precipitate with salts of magnesium. When freshly prepared it is colourless, or nearly so; on keeping, it becomes yellow from separation of  $\text{S}$  and formation of  $\text{Am}^2\text{S}^2$ ; by long keeping and exposure it becomes colourless, and is then useless.



#### SULPHINDIGOTIC ACID—INDIGO.



Pure indigo is warmed for an hour with fifteen parts of  $\text{H}^2\text{SO}^4$ , diluted with much water and filtered.

#### ACETIC ACID.



The strongest official acid is available, often contains traces of  $\text{H}^2\text{SO}^4$ , and sometimes  $\text{HCl}$ , detected as above by  $\text{BaCl}^2$  or  $\text{AgNO}^3$ .

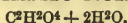


## TARTARIC ACID.



The official acid is sufficiently pure. The solution should be made by dissolving one part of acid in about six of water; the solution will not keep for more than a month or two, and must be renewed. The formation of a fungoid substance indicates the decay.

## OXALIC ACID.



The official acid is sufficiently pure. Dissolve one part of acid in ten of water; the solution is said to decompose by keeping, but this does not occur to such an extent as to interfere with any but the most delicate quantitative experiments.

## AMMONIUM OXALATE.

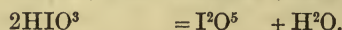
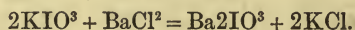
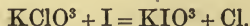


Obtained by neutralising  $H^2C^2O^4$  by  $NH^3$ , evaporating and crystallising. One part in ten or twelve of water. This solution has lately been stated to undergo decay, but its use may be dispensed with by using  $NH^3$  and  $H^2C^2O^4$ .

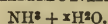
## IODIC ACID.



This reagent is useful in detecting  $SO^2$  and morphine, but must generally be prepared by the analyst. Digest equal parts of  $KClO^3$  and  $I$ , with eight parts of  $H^2O$ , to which a few drops of  $HNO^3$  have been added, until all the  $I$  has disappeared; of course only a gentle heat should be employed to prevent loss of  $I$ . Add  $BaCl^2$  till no more precipitate is formed, throw on filter, wash, dry. Digest the dried precipitate with one-fifth of its weight of  $H^2SO^4$ , dilute, filter, evaporate filtrate till solid mass of  $I^2O^5$  is obtained. It is best not to keep a solution of this reagent, as it is rather apt to be decomposed.



## AMMONIA.



The official preparation is generally very pure; should be diluted with one or two parts of water. In some cases excessively dilute

solutions are required, as in testing for nickel, cobalt, cadmium, &c. The solution acts usually as if it contained  $\text{Am}^2\text{O}$  like  $\text{K}^2\text{O}$ , or perhaps  $\text{HAmO}$  like  $\text{HKO}$ .


#### POTASSA—POTASSIUM HYDRATE.

##### $\text{HKO}$ .

The commercial article is generally very impure, containing chloride, sulphate, carbonate, silicate, and sometimes aluminate. The first two are detected by neutralising dilute solution with  $\text{HNO}^3$ , and testing for  $\text{HCl}$  and  $\text{H}^2\text{SO}^4$  by  $\text{AgNO}^3$  and  $\text{BaCl}^2$ ; the third by effervescence on addition of  $\text{HNO}^3$ . The latter two are present generally in only small proportions. A tolerably pure preparation can be made by boiling the official  $\text{HKO}$  in pure alcohol, evaporating the clear liquid to dryness and dissolving residue in water.

#### SODA—SODIUM HYDRATE.

##### $\text{HNaO}$ .

 The commercial soda-lye is often quite as pure as the above, and may be rendered still purer by the use of alcohol as described. They are both, however, available for ordinary purposes, but the presence of impurities must be borne in mind. A tolerably pure solution of  $\text{HNaO}$  may be obtained by mixing in a sufficiently large bottle 14 oz. of crystallised  $\text{NaCO}^3$ , 8 oz. freshly slaked lime, and 40 oz. of water; allowing to stand for twenty-four hours with frequent shaking; pouring off the clear liquid.

#### AMMONIUM CARBONATE.

##### Generally $\text{AmHCO}^3 + \text{Am}^2\text{CO}^3$ .

The official salt is generally very pure; dissolve in about ten parts of water at a gentle heat and add one-fourth of its bulk of dilute  $\text{NH}^3$ .

#### SODIUM CARBONATE.

##### $\text{Na}^2\text{CO}^3$ .

The crystallised carbonate (washing soda)  $\text{Na}^2\text{CO}^3 + 10 \text{H}^2\text{O}$  is generally impure, containing chloride and sulphate, detected as in  $\text{HKO}$ . The bicarbonate (baking powder) is usually much purer. By heating this for some time to dull red heat, the carbonate will remain sufficiently pure for most purposes. Dissolve in ten parts water.



## CALCIUM HYDRATE.

LIME WATER,  $\text{CaH}_2\text{O}_2$ .

Freshly burnt lime, best from marble, is slaked by addition of small quantity of water, and then soaked in a large quantity. The first portion of solution should be thrown away; a fresh quantity being poured on, is allowed to stand for some hours with occasional stirring, and then filtered.

## CALCIUM CHLORIDE.

 $\text{CaCl}^2 + \text{xH}^2\text{O}$ .

This may be obtained of sufficient purity by dissolving white marble in pure  $\text{HCl}$ , evaporating to dryness, and dissolving the salt in ten parts of water;  $\text{CaCO}^3 + \text{H}^2\text{Cl}^2 = \text{CaCl}^2 + \text{CO}^2 + \text{H}^2\text{O}$ . The solution should give no precipitate or even colour with  $\text{NH}^3$  or  $\text{Am}^2\text{S}$ , and should not react acid, as shewn by blue litmus paper.

## BARIUM CHLORIDE AND NITRATE.

 $\text{BaCl}^2 + 2\text{H}^2\text{O}$ ;  $\text{Ba}^2\text{NO}^3$ .

The official preparations are sufficiently pure, if recrystallised. They sometimes contain lead, which is indicated by brown colour or precipitate produced by  $\text{H}^2\text{S}$ . The solutions in fifteen or twenty parts of water may be treated with  $\text{H}^2\text{S}$ , allowed to stand in warm place for some hours, or boiled and filtered.

## POTASSIUM CYANIDE.

 $\text{KCy}$ .

Photographers' salt is sufficiently pure; solution, in ten parts water, cannot be kept for any length of time. It must be remembered that the commercial salt often contains chloride, cyanate and carbonate.

## POTASSIUM FERROCYANIDE.

 $\text{K}^4\text{Cfy} + 3\text{H}^2\text{O}$ .

Commercial salt is sufficiently pure. Dissolve in ten parts of water.

## POTASSIUM FERRICYANIDE.

 $\text{K}^3\text{Cfy}$ .

This salt not being always purchasable, may be prepared as follows: Through a solution of the yellow ferrocyanide in ten parts of water, chlorine gas is passed for some time until no more is absorbed. The solution should be contained in a bottle which it only half fills,

and every now and then the tube delivering the gas should be removed, and the bottle, being closed by the thumb, violently agitated. This must be continued until a drop of the solution gives no blue colour or precipitate with  $\text{Fe}^2\text{Cl}^6$ , but only a brown colour. This trial is best made by putting a few drops of the  $\text{Fe}^2\text{Cl}^6$  solution on a glass plate, and testing the fluid in the bottle from time to time, by taking out a drop on the end of a glass rod and touching the  $\text{Fe}^2\text{Cl}^6$ . When only a brown colour is produced, evaporate the solution till the beautiful red salt crystallises. The solution decomposes on keeping, and consequently must be made afresh now and then.

The chlorine required is best evolved from the following mixture : five parts  $\text{H}^2\text{SO}^4$ , four  $\text{H}^2\text{O}$ , three  $\text{NaCl}$ , two  $\text{MnO}^2$ , gently warmed in a flask provided with doubly bent tube. If the gas evolved from this mixture be conducted into *cold* water it will form another re-agent often required,  $\text{Na}^2\text{Cl}^2 + \text{MnO}^2 + 2\text{H}^2\text{SO}^4 = \text{Cl}^2 + \text{Na}^2\text{SO}^4 + \text{MnSO}^4 + 2\text{H}^2\text{O} + \text{K}^4\text{Cfy} + \text{Cl} = \text{K}^4\text{Cfy} + \text{KCl}$ .

#### CHLORINE WATER.



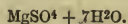
The same can be obtained very expeditiously, but not quite pure, by acting on  $\text{KClO}^3$  with  $\text{HCl}$ , and passing gas into water. The solution will not keep. A few drops of bromine shaken with water will form a solution equally available in most cases.

#### POTASSIUM SULPHOCYANATE.



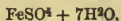
This salt is often not procurable from the drug store. Dry the ferrocyanide perfectly, take forty-six parts of the dry salt, seventeen dry potassium carbonate, thirty-two of sulphur. Fuse the mixture in a crucible at a dull red heat till the mass is liquid, fusing quietly. Pour out and dissolve the cooled mass in water, or preferably extract the powdered mass with alcohol. Dissolve in about ten parts of water. A solution of  $\text{KCyS}$  can be prepared by boiling  $\text{KCy}$  with sulphur for some hours, adding a little  $\text{HCl}$  to decompose  $\text{K}^2\text{S}$ , and filtering. The salt itself also, by fusing in an iron pot equal parts of  $\text{KCy}$  and  $\text{S}$ , extracting with proof spirit, &c.

#### MAGNESIUM SULPHATE.



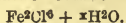
Commercial salt is sufficiently pure. Dissolve in ten parts of water.

## FERROUS SULPHATE.



Best obtained from residue of preparation of  $\text{H}_2\text{S}$ ; solution is boiled, filtered, evaporated rapidly and allowed to crystallise. Crystals should be rapidly dried, and preserved in well corked bottles. A more permanent salt is obtained by adding alcohol to above solution, filtering precipitate, washing with alcohol and drying. The ammonium-ferrous sulphate is also more permanent.

## FERRIC CHLORIDE.



Can be obtained by dissolving ferric oxide in  $\text{HCl}$ ; a better method, by which a purer preparation is obtained and which saves the trouble of making the oxide, is to digest clean iron nails with  $\text{HCl}$  as long as any action takes place, pour the liquid into a rather large bottle and pass chlorine through, as described under Potassium Ferricyanide, until a drop of the solution gives no blue, but only a brown colour with that reagent. The solution thus obtained should be boiled to expel excess of chlorine, and perhaps evaporated to syrup to remove hydrochloric acid. Dissolve in water.  $\text{Fe} + \text{H}^2\text{Cl}^2 = \text{FeCl}^2 + \text{H}^2$   
 $2\text{FeCl}^2 + \text{Cl}^2 = \text{Fe}_2\text{Cl}_6$ .

## SILVER NITRATE.



The salt used by photographers is generally quite pure. Dissolve in ten parts of water.

## LEAD ACETATE.



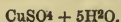
The commercial salt should be recrystallised, dissolved in ten parts of water, and filtered.

## MERCURIC CHLORIDE.



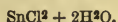
Dissolve in twenty parts or more of water.

## CUPRIC SULPHATE.



Recrystallise commercial salt, and dissolve in ten parts of water. The salt obtained from the residue left after preparation of  $\text{SO}^2$  is generally very pure. The copper used for military caps, being very free from all impurities, should be employed when procurable.

## STANNOUS CHLORIDE.



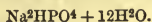
The salt used by dyers may be dissolved in ten parts of water, with addition of a few drops of HCl, filtered if necessary; or granulated tin may be digested with HCl. The operation is tedious if conducted in glass or porcelain vessels, but proceeds rapidly in vessels of copper, or even in glass, on addition of a few drops of  $\text{PtCl}^4$ , the precipitated platinum remains undissolved.

## PLATINIC CHLORIDE.



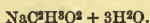
Dissolve scraps of platinum in a mixture of one part  $\text{HNO}^3$  and three HCl; evaporate to syrup and dissolve in a small quantity of water. The auric chloride  $\text{AuCl}^3$  can be obtained in the same way, or purchased from the photographers, but is not absolutely required.

## SODIUM PHOSPHATE.



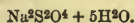
The commercial salt can be recrystallised and dissolved in ten or fifteen parts of water, but it generally contains a little sulphate. The medicinal phosphoric acid is usually pure, and by neutralising with pure  $\text{NaHCO}^3$ , a better preparation can be obtained.

## SODIUM ACETATE.



Can be purchased or made by neutralising  $\overline{\text{Ac}}$  with  $\text{NaHCO}^3$  and crystallising. Dissolve in ten parts of water. For some experiments it is well to drive off the water of crystallisation at a moderate heat.

## SODIUM HYPOSULPHITE.



The salt used by photographers is quite pure, and should be dissolved in ten parts of water.

## POTASSIUM CHROMATE.



The commercial bichromate dissolved in water is carefully neutralised with HKO until distinct alkaline reaction is perceptible, evaporated to crystallisation, and the yellow salt dissolved in twenty parts of water. In almost all cases the bichromate may be used instead.

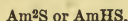


## CALCIUM SULPHATE.



Prepare the solution by means of gypsum (plaster of Paris) exactly in the same way as lime water from lime.

## AMMONIUM SULPHIDE.



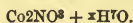
See under Hydrosulphuric Acid.

## POTASSIUM NITRITE.



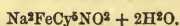
Warm gently some arsenious acid, preferably in lumps with  $\text{HNO}_3$ , and pass the gas into a solution of one part of  $\text{HKO}$  in forty of water as long as the gas is absorbed. This reagent is not absolutely required, but is useful in separating cobalt from nickel, and is a good test for the former.

## COBALT NITRATE.



Salts of cobalt are not often procurable at the drug stores—the so called cobalt is arsenic. By fusing for half an hour finely pounded smalt with four parts of  $\text{Na}_2\text{CO}_3$ , or preferably  $\text{NaKCO}_3$ , and boiling the fused mass repeatedly with water, cobalt oxide will remain, which can be dissolved in  $\text{HNO}_3$ ; only a small quantity of this reagent is required.

## SODIUM NITROPRUSSIDE.



Two parts  $\text{K}^4\text{Cfy} + 3\text{H}_2\text{O}$ , five parts  $\text{HNO}_3$ , with equal bulk of water, gently warmed under flue until gases cease to be evolved, and liquid gives no blue, but slate colour, with  $\text{FeSO}_4$ . Allowed to cool, liquid poured off from crystals of  $\text{KNO}_3$ , neutralised with  $\text{NaCO}_3$ , filtered, evaporated to crystallisation, and red crystals of nitroprusside separated mechanically, or preferably, solution evaporated to dryness, and treated with hot weak alcohol (50 per cent.), which extracts the nitroprusside. This reagent may be dispensed with, as it is rather difficult to prepare.

## POTASSIUM IODATE.

This salt is useful in distinguishing between spots or stains produced by  $\text{AsH}_3$  or  $\text{SbH}_3$ . Can be obtained by neutralising iodic acid with  $\text{K}_2\text{CO}_3$ , or in first part of preparation of iodic acid, as soon



as all iodine has disappeared, allowing mixture to cool.  $\text{KIO}^3$  separates out, can be washed with little water, not being very soluble.

#### ALCOHOL.

$\text{C}^2\text{H}^5\text{O}$ .

This must be strong, but need not be absolute. It must be pure, as methylated spirit is often not available except for burning in lamps; being so much lower in price, it is preferable for this purpose.

#### NESSLER'S SOLUTION.

Thirty-five grms. of  $\text{KI}$  and 13 grms. of  $\text{HgCl}^2$  are dissolved in about 800 CC of water heated, and a saturated solution of  $\text{HgCl}^2$  added until the precipitate formed ceases to be dissolved. 100 grms. caustic potassa are then dissolved in the liquid, and the whole made up to one litre.

#### SPECIAL TESTS.

##### INTRODUCTION.

The student having traced the base or acid in the salts, soluble in water with or without the aid of a few drops of nitric acid, should then take up the special tests, in order to render himself familiar with the general behaviour of the substance under examination. The Table Group and Division Tests need not of course be repeated unless where, as in some cases, there are certain reactions not mentioned therein. The tables for bases are perfectly satisfactory, those for acids are not quite so; in case of a difficulty, the Supplementary Table may be used. There is not unfrequently a doubt with beginners in testing for oxalates, ferro, ferri and sulpho cyanides, owing to most oxalates darkening only slightly, the ferro and ferri cyanides browning, and the sulpho cyanides, in some cases, scarcely darkening at all. In such cases the above mentioned table will be found useful, as also when a solution and not a dry salt is under examination.

At the suggestion of a friend engaged in teaching, the writer hereby appends a few remarks on the use of Table I., shewing the decompositions taking place, in addition to the instructions contained in page 1.

GROUP I.—When  $\text{HCl}$  is added to a solution of either  $\text{Pb}$ ,  $\text{Ag}$  or  $\text{Hg}$ , a chloride is formed insoluble in  $\text{H}^2\text{O}$  and in dilute acids.  $\text{PbO} + \text{H}^2\text{Cl}^2 = \text{PbCl}^2 + \text{H}^2\text{O}$ . When added to tartrate of antimony and

potassium (tartar emetic),  $\text{SbCl}^3$  is formed, which with  $\text{H}^2\text{O}$  is decomposed.  $3\text{SbCl}^3 + 3\text{H}^2\text{O} = \text{SbCl}^3\text{Sb}^2\text{O}^3 + 6\text{HCl}$ , which white precipitate is soluble in excess of  $\text{HCl}$ . When  $\text{HCl}$  is added to sodium tungstate,  $\text{NaCl}$  is formed and tungstic acid precipitated, which dissolves in excess of  $\text{HCl}$ . If zinc or tin be added to this solution, the acid is reduced to tungsten oxide, which has a blue colour. (Table II.) Similar action of  $\text{Zn}$  on molybdic acid.

GROUP II.—The addition of  $\text{H}^2\text{S}$  to the above acid solution causes the precipitation of such metallic sulphides as are insoluble in dilute acids. Some of them form soluble double sulphides with  $\text{Am}^2\text{S}$ ,  $\text{K}^2\text{S}$ , &c., such as  $\text{SnS}^2\text{K}^2\text{S}$ ,  $\text{As}^2\text{S}^3\text{K}^2\text{S}$ , &c.; others do not, such as  $\text{CdS}$ ,  $\text{CuS}$ . Hence the use of  $\text{Am}^2\text{S}$  in dividing this group into two parts. (Tables III. and IV.) A white precipitate or milkiness is formed by  $\text{Fe}^2\text{O}^3$ , the decomposition should be  $\text{Fe}^2\text{O}^3 + \text{H}^2\text{S}^3 = \text{Fe}^2\text{S}^3 + \text{H}^2\text{O}^3$ , but  $\text{Fe}^2\text{S}^3$  is unstable, and resolves itself into  $\text{Fe}^2\text{S}^2 + \text{S}$ , which causes milkiness, and  $\text{Fe}^2\text{S}^2$  dissolves in the free acid.  $\text{Fe}^2\text{S}^2 + \text{H}^4\text{Cl}^4 = \text{Fe}^2\text{Cl}^4 + \text{H}^2\text{S}^2$ . With chromic acid,  $\text{Cr}^2\text{O}^6 + \text{H}^2\text{S}^3 = \text{Cr}^2\text{O}^3 + \text{H}^2\text{O}^3 + \text{S}^3$ , presence of  $\text{Cl}$  or  $\text{Br}$ , or even  $\text{SO}^2$ , might produce the same result. An opalescence is not a precipitate.

GROUP III.—The sulphides of these metals being soluble even in dilute acids (unless it be a weak one such as acetic), no precipitate is formed by  $\text{H}^2\text{S}$  in acid solutions. Even without the addition of  $\text{HCl}$ , no precipitate is formed by  $\text{H}^2\text{S}$  if the metal is united to a strong acid.  $\text{FeSO}^4 + \text{H}^2\text{S}$  may be supposed to give  $\text{FeS} + \text{H}^2\text{SO}^4$ , but  $\text{FeS}$  is soluble in  $\text{H}^2\text{SO}^4$ , reproducing  $\text{FeSO}^4$  and  $\text{H}^2\text{S}$ . This sulphide is very easily soluble in even dilute  $\text{HCl}$ , while those of  $\text{Ni}$  and  $\text{Co}$ , when once formed, are not so easily dissolved; hence use of  $\text{HCl}$  in Table V.

In the case of  $\text{Cr}$  and  $\text{Al}$ , as the sulphides of these metals cannot be formed in the wet way, the precipitate is either  $\text{Cr}^2\text{O}^3$  or  $\text{Al}^2\text{O}^3$ , caused by the  $\text{NH}^3$ . Even  $\text{Am}^2\text{S}$  alone will produce the same effect.

GROUP IV.—No precipitate by  $\text{H}^2\text{S}$ , the sulphides being soluble in  $\text{H}^2\text{O}$ . The precipitate formed by  $\text{Na}^2\text{CO}^3$  is a carbonate.  $\text{BaCl}^2 + \text{Na}^2\text{CO}^3 = \text{BaCO}^3 + \text{Na}^2\text{Cl}^2$ . If much  $\text{AmCl}$  be present and  $\text{Am}^2\text{CO}^3$  be used,  $\text{MgCO}^3$  will not be precipitated, being soluble in salts of ammonia. Hence separation of  $\text{Mg}$  from  $\text{Ba}$ ,  $\text{Sr}$  and  $\text{Ca}$ , as in Table VI.

GROUP V.—The use of  $\text{PtCl}_4$  in Table VII. depends on the formation of difficultly soluble double chlorides,  $\text{K}^2\text{Cl}^2 + \text{PtCl}_4$ . The salts with Na and Li are soluble. On the same suggestion I have added formulas in some cases to the reactions contained in the following pages.

The experiments on testing may be made with very small quantities of the original solution in test tubes, or in many cases with a drop or two on a plate of glass. Where test tubes are not easily procurable, a large number of the experiments may be made in small trays made out of squares of paper, the edges doubled up. This simple contrivance (see Faraday's Manipulations) may be used, with a great saving of expense, but of course is not applicable where strong acids or heat are required; although, with a little care, liquids may be boiled in such a paper test dish. Strips of glass, such as employed for microscope slides, but of inferior quality, are quite available; they may be held in a clothes line clip if application of heat is necessary. Good test tubes, as above described, should, however, be procurable at the price of three, or, at the outside, four dollars per gross.

### PRELIMINARY EXAMINATION.

This table is placed here instead of before the preceding pages, as the writer's long experience is that it is not fitted for beginners, who are apt to jump at conclusions which are often erroneous. After having worked through the preceding tables, and acquired a certain amount of knowledge, dexterity in manipulation, and judgment, then the preliminary experiments may be most useful, and save much time and trouble. The table is taken, with several alterations, from Will's, and applies only to solids. A point occurs in which many beginners are wrecked, viz., the difference between *decrepitation* and *defflagration*. The first is caused by the conversion into steam of the small particles of water existing in many salts, which causes the substance to fly away in small fragments when heated on charcoal in the blowpipe flame; notable instances are  $\text{NaCl}$ ,  $\text{KI}$ ,  $\text{Pb}_2\text{NO}_3$ . To avoid this the substance should be powdered and moistened with water before heating, or the operation performed in a tube. Any broken old test tubes may be sealed up, and are quite available for this purpose. Residue remaining in tube, if no blackening has taken place, can then be used for shewing defflagration. The second causes vivid combustion of the charcoal; notable instances, nitrates and chlorates.



## PRELIMINARY EXAMINATION OF SOLIDS,

## WITH OR WITHOUT USE OF BLOWPIPE.

Colour.	Chromates, red, yellow, brown. Cobalt, pink, red, blue. Manganese, pink, purple, green. Some iodides, red, yellow, green. Chromitum, purple, green, black.				
Having metallic lustre.	Metals and many metallic sulphides. Iodine.				
Fusible on C without change.	Salts of alkalis and alkaline earths; residue is often alkaline, as shewn by browning of turmeric paper. Some silicates.				
<del>Fusible on C with incrustation.</del>	Some metals or their compounds, such as Zn, Cd, Pb, Sb, white Sb, yellow when hot Zn, brownish Cd, reddish yellow Pb.				
Acquiring darker colour.	Organic salts. Many oxides, zinc, yellow while hot. Mercuric oxide, brown or black when hot. Lead oxide, red Hg <sup>12</sup> , yellow, turning red when rubbed.				
Colouring outer flame of BP.	Violet K, yellow Na, dark crimson Li, crimson Sr, green Ba Cu, green blue Cu, orange Ca, &c.				
Heated on C with Co <sub>2</sub> NO <sub>3</sub> .	Some phosphates, borates and silicates.	Blue Glass.	Blue Mass.	Green Mass.	Zinc oxide and salts.
<del>Heated on C with Na<sub>2</sub>CO<sub>3</sub>, producing</del>	Odour of Garlic.	Hepar.	Metallic grains without sublimate,	Metallic globule with sublimate,	Sublimate without metallic globule, Zn white, Cd brown.
	Arsenic.	All compounds of sulphur.	Sr, Ag, Cu, Au.	Sb, Bi, Pb.	
Heated in sloping open tube, producing	Odorous Gases.	Metallic Sublimate.	White Sublimate.	Water.	Hydrates and many bodies.
	Sulphides, arsenides, some salts of Am.	As, Hg compounds.	Crystalline, As, amorph. Hg, Am.		
Deflagrating when heated on charcoal.	Nitrates, chlorates, iodates, bromates, perchlorates, &c.	Lead and bismuth oxides also exhibit the property in a minor degree.			
With zinc and HCl giving	Violet colour.	Blue, finally black.	Blue, finally red.	Green.	Chromium as chromic acid.
	Titanium.	Molybdenum.	Trungsten.		

## BLOWPIPE TESTS

*With Beads of Borax on Platinum Wire, or Phosphor (microcosmic) Salt on Wire or Charcoal.*

To save the expensive platinum wire, it is well to fuse one end of a piece about two inches long into a short glass tube drawn out; this serves as a handle, and as only a very small piece of the wire need be fused into the glass, the wire can be used to the last quarter inch.

In the same way a glass handle can be fused on to a small piece of platinum foil, too short to be held in the blowpipe flame.

The use of the blowpipe cannot be sufficiently recommended, as it often gives a clue at once to the substance under examination, and is constantly employed as a means of confirmation. A spirit lamp is not well adapted for experiments, but if filled with a mixture of eight or ten parts of alcohol to one of oil of turpentine, and having a rather large wick, if flattened instead of round it will be preferable, and be found to answer all requirements. For holding the wick a thin tube may be used, fastened into a cork which fits into any common drug bottle; the end of the tube should be pressed flat, so as to allow a flat wick to pass through, such as used in our ordinary lamps. The top of the tube, and of course the wick, should be cut obliquely, and the current of air from the blowpipe driven downward parallel to the wick. With a little ingenuity, one of our small coal oil lamps may be arranged so as to suit admirably.

The mode of keeping up a continuous blast can scarcely be explained—it is an art that can only be learnt by practice; but the following remarks (condensed), extracted from the admirable work of Harcourt and Madan, seem to offer the best explanation. “There are two essential parts in a double organ bellows: the first, the feeder (lungs), which supplies the air; the second, the reservoir (mouth), which stores up the air received from the feeder and supplies it as required to the organ pipes. There must also be a valve (tongue) between the feeder and reservoir, and also weights or force (muscles of cheeks) acting on the air to drive it out. When we distend the cheeks with air from the lungs, it is easy to breathe through the nose, but the difficulty is, when sending air through the blowpipe, to use the tongue as a valve to admit air from the nose without interrupting the blast. A little practice will soon



overcome the difficulty of balancing the action of the lungs and of the cheeks."

When a steady blast can be obtained, the blowpipe flame should exhibit a blue cone surrounded or ended by a more faintly luminous portion; the blue is the reducing, the outer the oxidising portion. To get the reducing flame, the light should be large, whether from gas or oil, the tip of the blowpipe introduced *just* into the luminous portion, and the blast not very strong.

To get the oxidising flame in perfection, the light should be smaller, the tip of blowpipe introduced further into the luminous cone, and the blast stronger. The hottest part is just at the end of the blue cone. A good proof of being able to keep up a good reducing flame is to melt a small piece of tin on charcoal, and keep the globule metallic; also to decolorise a well coloured amethyst bead, formed by manganese, to a colourless one. A very small portion of tin-foil added to the fusing borax greatly assists the reduction, but is almost sure to destroy the platinum wire.

The bead of borax or phosphor is apt to fall off, if the loop in the wire (made by winding it round a glass rod) is too large; the difficulty may be got over by making the loop smaller, or by roughening the surface of the platinum by placing it for a few moments in hot aqua regia; also by other methods which are not generally available. In testing any substance in a bead, *very* small quantities should be added at first, and gradually increased till the desired result is obtained; and the wires after use may be soaked in dilute HCl to remove the bead, then in pure water, the operation being repeated until no colour is noticed on heating the wire in the blow-pipe flame. The writer has noticed that wires used for shewing the fine green flame of barium are very hard to clean; after constant soaking, or even boiling, the green colour comes out.

#### USE OF BORAX BEAD IN BLOWPIPE EXPERIMENTS.

When heated in oxidising flame (OF) the bead formed on loop of platinum wire should be held just outside the cone of the OF, for reducing flame RF just beyond the blue cone but well in the outer cone. The process called flaming consists in submitting the bead to intermittent puffs of flame. If colour be too dark, the bead may be flattened in pincers while still hot.

	OF	RF
Mn....	Violet .....	Colourless if quickly cooled.
Fe ....	Yellow or reddish .....	Bottle green.
Cr ....	Yellow red, while hot.....	Emerald green.
	Greenish, when cold .....	
Ni ....	Violet brown, hot.....	Gray and opaque.
	Clear brown, cold.....	
Co ....	Blue .....	Blue.
Cu ....	Green, hot.....	Colourless while hot.
	Greenish blue, cold .....	Opaque red when cold.
Ti ....	Yellow, hot .....	Yellow or brownish.
	Colourless, cold .....	Opaque light blue by flaming.
	Opaque by flaming.	
Tu ....	Same as Ti.....	Same as Ti.
Mo....	Yellow, hot .....	Brown or gray, semi-opaque, often
	Colourless or yellow, cold .....	with black specks.
	Grayish opaque by flaming.	

Pb, Bi, Ag, Sb, Al, Sr, Sn, give no very characteristic reactions; see former page. Ba, Sr, Ca, give beads opaque on cooling or by flaming. Mo, gives with phosphor salt, on cooling, a fine green; Tu a greenish blue; if Fe be present, a blood red or brownish colour. Ti, of pure violet; when cold, if Fe be present, same as Tu.

The above directions are copied (with omissions) by permission, from Prof. Chapman's work on Canadian minerals.

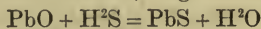
## TABLE II.—GROUP I.

### LEAD.

1. HKO produces a white precipitate soluble in excess. A very small quantity of the precipitate should be used, as PbO is only slightly soluble in HKO.  $\text{Pb}_2\text{NO}^3 + 2\text{HKO} = \text{H}^2\text{PbO}^2 + 2\text{KNO}^3$ .

2.  $\text{NH}^3$  produces a white precipitate, insoluble in excess. Except in the case of the commonly occurring lead acetate (sugar of lead), in which  $\text{NH}^3$  produces no precipitate unless used in large excess. Same formula, substituting  $\text{NH}^4$  for K.

3.  $\text{H}^2\text{S}$  produces black precipitate, insoluble in dilute acids and alkalic sulphides. The dry or partially dry precipitate is converted into white  $\text{PbSO}^4$  by the action of  $\text{HNO}^3$  when heated. Fused on charcoal by itself, or with  $\text{Na}^2\text{CO}^3$ , it gives malleable beads of the metal.



4. HCl produces white precipitate, soluble in excess of boiling water; the solution on cooling deposits  $\text{PbCl}^2$  in small crystals.  $\text{PbCl}^2$  being partially soluble in water, must be looked for in both Groups I. and II.  $\text{PbO} + \text{H}^2\text{Cl}^2 = \text{PbCl}^2 + \text{H}^2\text{O}$ .

5. KI produces a bright yellow precipitate, soluble in large amount of boiling water; on cooling, is deposited in golden scales.  $\text{Pb2NO}^3 + \text{K}^2\text{I}^2 = \text{PbI}^2 + 2\text{KNO}^3$ .

6.  $\text{K}^2\text{CrO}^4$  produces yellow precipitate, soluble in large quantity of HKO; only small quantity of precipitate to be used.  $\text{Pb2NO}^3 + \text{K}^2\text{CrO}^4 = \text{PbCrO}^4 + 2\text{KNO}^3$ .

7.  $\text{H}^2\text{SO}^4$  or any sulphate produces white precipitate of  $\text{PbSO}^4$ , insoluble in water or dilute acids, soluble in hot HCl, and AmAc.  $\text{Pb2NO}^3 + \text{H}^2\text{SO}^4 = \text{PbSO}^4 + 2\text{HNO}^3$ .

8. Salts of lead heated with  $\text{Na}^2\text{CO}^3$  on charcoal, with or without KCy, give malleable beads of lead.  $\text{PbO} + \text{C} = \text{Pb} + \text{CO}$ , or  $\text{Pb}^2\text{O}^2 + \text{C} = \text{Pb}^2 + \text{CO}^2$ .

#### SILVER.

1. HKO produces a brown precipitate, insoluble in excess, but soluble in  $\text{NH}^3$ .  $2\text{AgNO}^3 + 2\text{HKO} = \text{Ag}^2\text{O} + 2\text{KNO}^3 + \text{H}^2\text{O}$ .

2.  $\text{NH}^3$ , VERY dilute, produces the same coloured precipitate, soluble in excess; the  $\text{NH}^3$  must be *very* dilute.

3.  $\text{H}^2\text{S}$  produces a black precipitate, insoluble in dilute acids, but soluble in strong  $\text{HNO}^3$ . In this, as in all following cases, the precipitate should be allowed to settle, often hastened by shaking or warming, the liquid poured off, and then the test applied. In some, if not all cases, it is well to pour on some water, and repeat this washing several times.

4. HCl produces white precipitate, soluble in  $\text{NH}^3$ , KCy and  $\text{Na}^2\text{S}^2\text{O}^8$ . In using latter test, the precipitate must be well washed and freed from acid, otherwise an opalescence (sulphur),  $\text{Na}^2\text{SO}^3 + \text{H}^2\text{Cl}^2 = \text{Na}^2\text{Cl}^2 + \text{SO}^2 + \text{S} + \text{H}^2\text{O}$ , will be observed.  $\text{AgNO}^3 + \text{HCl} = \text{AgCl} + \text{HNO}^3$ .

5. Salts heated on charcoal with dry  $\text{Na}^2\text{CO}^3$  will give a brilliant bead of metal, not very malleable.

6. Addition of  $\text{FeSO}^4$  causes a gray precipitate of metallic silver in salts of that metal.

7. Salts of silver heated with neutral potassium tartrate, produce a black or metallic precipitate of the metal; the result is not readily produced by acid salts.



## MERCURY.

## MERCUROUS SALTS.

1. HKO produces a grayish black precipitate, insoluble in excess.  
 $2\text{HgNO}^3 + 2\text{HKO} = \text{Hg}^2\text{O} + 2\text{KNO}^3 + \text{H}^2\text{O}$ .

2.  $\text{NH}^3$  has the same effect, but the precipitate is not  $\text{Hg}^2\text{O}$ , but an ammoniacal compound.

3.  $\text{H}^2\text{S}$  produces a black precipitate, insoluble in  $\text{HCl}$  or  $\text{HNO}^3$ , soluble in a mixture of the two, forming a mercuric salt. It is soluble in a hot solution of  $\text{K}^2\text{S}$ ,  $\text{Hg}$  being separated.  $\text{Hg}^2\text{S} = \text{Hg} + \text{HgS}$ .

4.  $\text{HCl}$  produces a white precipitate, insoluble in excess, when boiled with strong  $\text{HCl}$ , turns gray from formation of  $\text{Hg}$  and  $\text{HgCl}^2$ .  $\text{Hg}^2\text{Cl}^2 = \text{Hg} + \text{HgCl}^2$ . By addition of  $\text{NH}^3$ , becomes gray or black from formation of  $\text{HgClHgNH}^2$ .  $\text{Hg}^2\text{Cl}^2 + \text{NH}^3 = \text{HgClHgNH}^2 + \text{HCl}$ .

5.  $\text{KI}$  produces a dull green precipitate of  $\text{HgI}$ .

6.  $\text{SnCl}^2$  produces a gray precipitate of  $\text{Hg}$ .  $\text{HgCl}^2 + \text{SnCl}^2 = \text{Hg}^2 + \text{SnCl}^4$ .

7. Solutions produce a gray stain on copper or gold, passing away on application of heat.  $\text{Hg}^2\text{NO}^3 + \text{Cu} = \text{Hg} + \text{Cu}^2\text{NO}^3$ .

8. Salts are all volatile, sometimes with deposition of  $\text{Hg}$ .

9. Salts heated in quill tube, with *perfectly* dry  $\text{Na}^2\text{CO}^3$ , give a sublimate of metallic  $\text{Hg}$ . If this be not easily recognised, the globules of  $\text{Hg}$  may be rendered more apparent by rubbing the sublimate with a piece of wood or quill.  $2\text{HgNO}^3 + \text{Na}^2\text{CO}^3 = \text{Hg}^2 + 2\text{NaNO}^3$ .

## GROUP I.

Mixture—Ag, Pb, Hg.

From a mixture of the chlorides, that of lead can be dissolved out by boiling with a large quantity of water, and the metal detected by  $\text{H}^2\text{SO}^4$ , or other appropriate tests. The residue, warmed with excess of  $\text{NH}^3$ , will yield up the  $\text{AgCl}$ , which can be reprecipitated from the filtered solution by  $\text{HNO}^3$ , and tested for  $\text{Ag}$  by fusion with  $\text{Na}^2\text{CO}^3$ , or other tests. The gray residue can be tested for  $\text{Hg}$  by heating in tube with  $\text{Na}^2\text{CO}^3$ , or other means.



## GROUP II.—TABLE III.

Sulphides Soluble in  $\text{Am}^2\text{S}$ .

## PLATINUM.

1. Soluble salts give, with salts of K acidulated with HCl, a difficultly soluble yellow precipitate,  $\text{PtCl}^4 \cdot 2\text{KCl}$ .
2. The same, with salts of  $\text{NH}^3$ ,  $\text{PtCl}^4 \cdot 2\text{AmCl}$ .
3. With  $\text{SnCl}^2$ , an intense brown colour,  $\text{SnCl}^2 + \text{PtCl}^4 = \text{SnCl}^4 + \text{PtCl}^2$ .
4. With KI, an intense brown or black colour,  $\text{PtI}^4$ .
- × 5. Metal soluble only in mixture of HCl and  $\text{HNO}^3$ ; not in either separately.

## GOLD.

1. Concentrated solutions give, with HKO and  $\text{NH}^3$ , yellow precipitate.
2.  $\text{SnCl}^2$ , if partially oxidized, gives purplish red precipitate or colouration.
3. Solutions warmed with oxalic acid give precipitate of gold, and blue colour.
4. With ferrous sulphate, a brown precipitate; or, if present in very small quantity, a blue colour. There must be no free  $\text{HNO}^3$  present, as that might produce brown colour.

*Addenda to Gold.*

Pure gold is not acted on by  $\text{HNO}^3$  or by HCl, but only by a mixture of the two.

Spurious gold, copper and zinc alloy, is acted on by  $\text{HNO}^3$  producing red fumes and green-blue solution.

Iron pyrites, often mistaken for gold, is, when powdered, acted on by hot  $\text{HNO}^3$ , with evolution of red fumes, general separation of sulphur and formation of a solution containing  $\text{Fe}^2\text{O}^3$  and  $\text{H}^2\text{SO}^4$ , detected by  $\text{NH}^3$  and  $\text{BaCl}^2$  after dilution.

Iron pyrites,  $\text{FeS}^2$ , when heated loses S, burning with blue flame and evolving  $\text{SO}^2$ , leaving FeS, which is fusible and attracted by magnet.

Gold can with difficulty be fused, and is not attracted by the magnet.

Native gold generally contains silver, easily extracted by  $\text{HNO}^3$  and recognized.

Coin and jewellers' gold generally contains copper, easily extracted and recognised.

The specific gravity of pure gold is 19.5; of coin gold, 17.5; of jewellers' gold, from 14 to 14.5. This is the safest way of determining the relative values of different kinds of so called golds. Spurious articles will have a specific gravity of 10 or less, and if under 14, the absolute quantity of genuine gold may be judged from following numbers, kindly supplied by Prof. Chapman. See addenda.

## ARSENIC.

### ARSENIOUS OXIDE OR ACID.

1. Acid and salts give no precipitate with HKO or  $\text{NH}^3$ .

2.  $\text{H}^2\text{S}$  produces no precipitate in solutions of the acid itself, but a bright yellow precipitate in acidulated solutions. Soluble in  $\text{NH}^3$ , HKO,  $\text{Am}^2\text{CO}^3$ , and  $\text{Am}^2\text{S}$ ; insoluble in dilute acids. Soluble in hot  $\text{HNO}^3$ , water having been removed. The solution in HKO, warmed with bismuth hydrate, turns it brown-black, and gives a solution of  $\text{As}^2\text{O}^3$  in HKO.  $\text{As}^2\text{S}^3 + \text{Bi}^2\text{O}^3 = \text{As}^2\text{O}^3 + \text{Bi}^2\text{S}^3$ . Heated repeatedly when dry with  $\text{HNO}^3$  gives residue of  $\text{As}^2\text{O}^3$  or  $\text{As}^2\text{O}^5$ , mixed of course with  $\text{H}^2\text{SO}^4$ . The dried sulphide fused with  $\text{KNO}^3$ , gives potassium arsenate. For detection of arsenic acid, see next page.

3. Solution of acid (only soluble to about two parts in one hundred) gives no precipitate with  $\text{CuSO}^4$ , till after careful addition of  $\text{NH}^3$ , bright green precipitate, soluble in least excess of acid to colourless solution, or in excess of  $\text{NH}^3$  to blue solution. The precipitate is Scheele's (Paris) green, and care must be taken to add only sufficient  $\text{NH}^3$  to produce the characteristically coloured precipitate. Salts of  $\text{As}^2\text{O}^3$  will of course produce it at once. This is sometimes called Scheele's Test, or Copper Test.

4. The green precipitate is soluble in HKO, and the solution when heated becomes opaque and red from separation of  $\text{Cu}^2\text{O}$ , cuprous oxide. In order to succeed in getting a clear blue solution in HKO, only a few drops of  $\text{CuSO}^4$  should be used; too much spoils the reaction.

5. The solution of  $\text{As}^2\text{O}^3$  gives, with  $\text{AgNO}^3$ , a white opalescence, but on careful addition of  $\text{NH}^3$ , a bright yellow precipitate, soluble

in least excess of acid, even acetic or  $\text{NH}^3$ ; hence care in adding  $\text{NH}^3$ . Hume's or Silver Test.

6. Solutions of  $\text{As}^2\text{O}^3$ , or its salts, mixed with a little  $\text{HCl}$ , and boiled with a clean strip of metallic copper, communicate to it a steel-gray stain, and, if strong, form steel-gray flakes. Reinsch's Test; see Dry Tests.

7. Solutions of  $\text{As}^2\text{O}^3$ , mixed with pure zinc and  $\text{H}^2\text{SO}^4$ , evolve  $\text{AsH}^3$ , which may be detected by causing the gas to issue from a narrow tube, and inflaming, it burns with a white light, and produces brown or metallic spots on a plate of porcelain (crucible cover) held in it. The gas is excessively poisonous.

8. Also by placing over the mouth of the tube or capsule in which the experiment is made a piece of paper moistened with  $\text{AgNO}^3$ ; a black or brown stain will indicate  $\text{AsH}^3$ . Fleitman's Test.

9. Almost any salt of  $\text{As}^2\text{O}^3$ , placed in a capsule with a little water, will, on the addition of sodium amalgam ( $\text{HgNa}$ ) in very small quantity, evolve enough  $\text{AsH}^3$  to darken a paper, moistened with  $\text{AgNO}^3$ , placed over it. Antimony will not produce the same effect. The same result by boiling solution of  $\text{As}^2\text{O}^3$  in  $\text{HKO}$ , with metallic zinc. The amalgam test is by Davy, and is most accurate.

✓ The above may be called the liquid tests for arsenic. The dry tests are as follows:

10. The dry acid or salt, heated on charcoal, or with  $\text{Na}^2\text{CO}^3$  and  $\text{KCy}$ , evolves vapour smelling of garlic.

11. (a) The dry acid or salt, mixed with  $\text{Na}^2\text{CO}^3$  and  $\text{KCy}$  (or charcoal), and heated in a narrow tube closed at one end, forms a sublimed ring, either brown or metallic.

(b) For examining the dry acid, place a portion of the substance in a closed tube, cover it with splinters of charcoal, heat the carbon red hot first in blowpipe flame, and then turn the tube so as to volatilise the  $\text{As}^2\text{O}^3$ ; a brown or metallic ring will be formed.  $\text{As}^2\text{O}^3 + \text{C}^3 = \text{As}^2 + \text{C}^3\text{O}^3$ .

12. (a) The lower end of the tube in which the ring has been formed may now be cut off with a file, the ring *very* gently warmed, and the garlic odour detected.

(b) The tube with ring may be held in an oblique position, and the ring *very* gently warmed, so as to allow a current of air to pass



over the heated portion. A white sublimate will be formed some distance above the heated spot.

(c) The sublimate is evidently crystalline, shewing under microscope octahedral crystals.

(d) To get better crystals, heat the bottom of the tube, then gently the crystalline ring. The vapours of  $\text{As}^2\text{O}^3$  being very heavy, will sink down and deposit themselves in distinct octahedral crystals, in the lower end of the tube, as it cools.

(e) The ring of As or  $\text{As}^2\text{O}^3$  may be washed out with warm  $\text{HNO}^3$ , evaporated in capsule to dryness, when  $\text{As}^2\text{O}^5$  will be formed, which gives a red-brown precipitate or colour with  $\text{AgNO}^3$ .

13. The strip of copper with steel-gray coating, obtained in test 6, should be taken out, washed, dried in blotting paper, cut into strips, introduced into a quill tube, and heated. A white crystalline ring is formed above the heated spot, from the As combining with the O of the air, forming  $\text{As}^2\text{O}^3$ .

14. Dry  $\text{As}^2\text{O}^3$ , heated with excess of dry sodium acetate, evolves the fearful odour of alkarsin,  $\text{C}^2\text{H}^6\text{As}$ . On account of its poisonous character, care must be taken in making this experiment. ✕

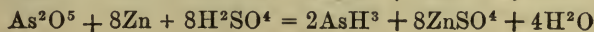
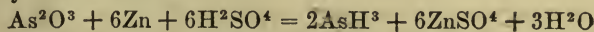
#### ARSENIC ACID.

1.  $\text{As}^2\text{O}^5$  is not precipitated by  $\text{H}^2\text{S}$  until after some time or by warming, it appears that the  $\text{As}^2\text{O}^5$  is reduced firstly to  $\text{As}^2\text{O}^3$ , and the yellow precipitate is  $\text{As}^2\text{S}^3 + \text{S}$ .

2.  $\text{AgNO}^3$  produces with salts of  $\text{As}^2\text{O}^5$ , and to some extent with the acid itself, a reddish brown precipitate of silver arsenate  $\text{Ag}^3\text{AsO}^4$ , not very easily soluble in  $\overline{\text{Ac}}$ ; hence can be separated from silver arsenite, and the presence of arsenic acid detected in old solutions of alkalic arsenites.

3.  $\text{NH}^3$  with  $\text{MgCl}^2$  gives a white precipitate with salts of  $\text{As}^2\text{O}^5$ , or with acid itself, soluble in  $\overline{\text{Ac}}$ , crystalline when slowly formed.  $\text{Na}^2\text{HAsO}^4 + \text{MgCl}^2 + \text{NH}^3 = \text{MgNH}^4\text{AsO}^4 + \text{Na}^2\text{Cl}^2$ , a reaction precisely similar to that with phosphates.

4. The tests with copper foil and zinc are the same as for arsenious acid.  $\text{AsH}^3$  is produced from acid solutions of arsenious and arsenic acids by zinc as follows :





## STANNIC SALTS.

1. Salts give with  $\text{HKO}$  a white precipitate, soluble in excess.
2. Same with  $\text{NH}^3$ , nearly insoluble in excess.
3. With  $\text{H}^2\text{S}$  a pale yellow precipitate, insoluble in  $\text{NH}^3$  if all  $\text{H}^2\text{S}$  has been previously removed, but easily soluble in soluble sulphides. Dried and treated with  $\text{HNO}^3$ , gives stannic oxide (metastannic acid).
4. Same as test 6 for stannous salts.
5. Metallic tin is converted by  $\text{HNO}^3$  into stannic oxide,  $\text{SnO}^2$ , and is dissolved by hot  $\text{HCl}$  and  $\text{HNO}^3$  to  $\text{SnCl}^4$ .

## ANTIMONY.

The usual salt employed in testing is tartar emetic, which, when in solution, gives a precipitate with  $\text{HCl}$ , soluble in excess. See Group I. This acid liquid must be regarded as the original solution. All salts of antimony not containing an organic acid are decomposed by water, giving white precipitates. Hence, if above original solution be diluted, a white precipitate will be formed, which must be dissolved up by a little  $\text{HCl}$ .

1.  $\text{HKO}$  produces precipitate, soluble in excess.
2.  $\text{NH}^3$  produces precipitate, insoluble in excess.
3. The orange precipitate with  $\text{H}^2\text{S}$  is soluble in  $\text{HKO}$ , and  $\text{Am}^2\text{S}$ , scarcely in  $\text{NH}^3$  or  $\text{Am}^2\text{CO}^3$ . Dried and fused with  $\text{KNO}^3$ , leaves on washing with hot water a residue of antimonate.
4. Solutions warmed with  $\text{HCl}$  and metallic copper communicate a gray metallic coating to the latter metal; this, on heating in tube, does not give octahedral crystals.
5. Acid solutions act on metallic zinc, forming a black precipitate or coating on the zinc, and evolving a gas which burns with a whitish flame,  $\text{SbH}^3$ . See under Poisons—Arsenic.
6. A drop of the acid solution placed on platinum foil and touched with a piece of zinc, produces a black stain, soluble in  $\text{Am}^2\text{S}$ .
7. Salts heated on charcoal with  $\text{Na}^2\text{CO}^3$ , give a brittle metallic bead and white incrustation.
8. The  $\text{SbH}^3$  produced in test 5 by zinc or by sodium amalgam, does not blacken paper soaked in  $\text{AgNO}^3$ .

Precipitate soluble in old  $\text{Am}^2\text{S}$ , or in new with addition of S.

### STANNOUS SALTS.

1. Salts give with HKO a white precipitate, soluble in excess.
2. Same with  $\text{NH}^3$ , but insoluble in excess.
3. The precipitate with  $\text{H}^2\text{S}$  is distinctly brown, and insoluble in fresh  $\text{Am}^2\text{S}$ ; by addition of S it is converted from  $\text{SnS}$  into  $\text{SnS}^2$ , and becomes soluble to yellow solution.
4. Salts give a purplish red precipitate with  $\text{AuCl}^3$ .
5. A white precipitate with  $\text{HgCl}^2$ , turning to gray on using excess and warming.
6. Heated on charcoal with  $\text{Na}^2\text{CO}^3$ , yield globules of soft metallic tin, which can be obtained in spangles of a white colour, as described under Copper.

### GROUP II.

Sulphides insoluble in  $\text{Am}^2\text{S}$ .

#### BISMUTH.

1. Salts give with HKO a white precipitate, insoluble in excess.  

$$2\text{Bi}3\text{NO}^3 + 6\text{HKO} = \text{Bi}^2\text{O}^3\text{H}^6\text{O}^3 + 6\text{KNO}^3.$$
2. With  $\text{NH}^3$  the same, substituting  $\text{NH}^4$  for K.
3. With  $\text{H}^2\text{S}$  a brown-black precipitate, soluble in warm strong  $\text{HNO}^3$ .
4. With  $\text{K}^2\text{CrO}^4$  a yellow precipitate, insoluble in HKO.
5. Salts heated on charcoal with  $\text{Na}^2\text{CO}^3$ , give a brittle bead of metal, and a brownish-yellow incrustation.
6. All salts not containing an organic acid are decomposed, and become opaque with water. They must be dissolved by heating with as little  $\text{HNO}^3$  as possible. 
$$2\text{BiCl}^3 + 3\text{H}^2\text{O} = \text{BiCl}^3 + \text{BiO}^3 + 6\text{HCl}.$$
 With large excess of acid this reaction does not succeed well. The excess of acid should be driven out by heating.
7. Very small quantities of bismuth may be detected by the dark colour given to the yellow lead iodide, or by heating the substance containing it with a little cupric iodide and sulphur; red iodo sulphide sublimes in crystals. See under Galena.

#### LEAD

Is introduced into this group as well as into I; the  $\text{PbCl}^2$  being partly soluble in  $\text{H}^2\text{O}$ , so that in very dilute solutions no precipitate would be formed by HCl.

## COPPER.

\* 1. HKO produces a pale blue precipitate,  $\text{CuOH}_2\text{O}$ , insoluble in excess, but turning black on heating,  $\text{CuO}$ .

2.  $\text{NH}^3$  produces the same, soluble in excess to a fine blue solution (characteristic test). The substance so formed may be considered, if the sulphate has been used, as a compound of  $\text{NH}$  therewith, or as the sulphate of an ammonium containing copper.

3.  $\text{H}^2\text{S}$  produces brownish-black precipitate, insoluble in dilute acids, soluble in  $\text{HNO}^3$  to blue solution; not quite insoluble in alkalic sulphides.

4.  $\text{K}^4\text{Cfy}$  produces brown-red precipitate,  $\text{Cu}^2\text{Cfy}$ , decomposed by HKO, giving blue hydrate, turning to black when heated.

5. Polished iron or steel, when introduced into a solution of  $\text{Cu}$ , becomes red from deposition of the metal.  $\text{CuSO}^4 + \text{Fe} = \text{FeSO}^4 + \text{Cu}$ .

6. Salts fused on charcoal with  $\text{Na}^2\text{CO}^3$  or  $\text{KNaCO}^3$ , with or without  $\text{KCy}$ , give a mass which, when scraped off, ground up in mortar with water, and the lighter particles washed away, exhibits small metallic spangles of copper.

7. Salts of  $\text{Cu}$  give a green or blue tinge to the alcohol or blowpipe flame. Copper pyrites may be easily recognised, by roasting a portion of the ore in the pincers or on charcoal, moistening with  $\text{HCl}$  and again applying the outer blowpipe flame; a blue colour will be observed. In examination of salts in this way, the use of  $\text{HCl}$  or of glycerine renders the results more apparent.

See Blowpipe Table for other tests.

## MERCURY.

## MERCURIC SALT.

1. HKO produces a brownish precipitate if added in very small quantity, but a bright yellow,  $\text{HgO}$ , if in excess. Insoluble in any amount of the precipitant. The brown precipitate is a compound, if  $\text{HgCl}^2$  be used, of  $\text{HgCl}^2$  with  $\text{HgO}$ ; if salts of  $\text{NH}^3$  are present, the precipitate is white.

2.  $\text{NH}^3$  produces a white precipitate, insoluble in excess.  $\text{HgCl}^2 + 2\text{NH}^3 = \text{HgH}^6\text{N}^4\text{Cl}^2$ . Mercuric ammonium chloride.

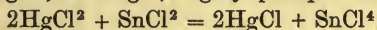
3.  $\text{H}^2\text{S}$  produces in gradually increasing quantities, especially with solutions of  $\text{HgCl}^2$ , a white, yellow, brown,  $\text{HgCl}^2 + x\text{HgS}$ , and lastly black precipitate, insoluble in  $\text{HCl}$  or  $\text{HNO}^3$ , soluble in a mixture of the two.



4. HCl produces no precipitate.

5. KI produces at first a yellow precipitate, turning to bright scarlet, soluble in excess of precipitant to a perfectly colourless solution.  $\text{HgI}^2\text{K}^2\text{I}^2$ . See Nessler's Test, and Alkaloids.

6.  $\text{SnCl}^2$  produces, when added to  $\text{HgCl}^2$  in small quantity, a white precipitate of  $\text{HgCl}$ ; in larger, a gray precipitate of metallic Hg.



The other tests are the same as for mercurous salts.

NOTE.— $\text{HgI}^2$  will *not* give sublimate of Hg when heated with  $\text{Na}^2\text{CO}^3$ , owing to volatility of  $\text{HgI}^2$ .

#### CADMIUM.

1. HKO produces a white precipitate, insoluble in excess.

2.  $\text{NH}^3$  produces a white precipitate, easily soluble in excess; hence weak  $\text{NH}^3$  should be employed.

3. The bright yellow precipitate formed by  $\text{H}^2\text{S}$  is insoluble in  $\text{NH}^3$  or  $\text{Am}^2\text{S}$ , also in dilute acids, if cold; but soluble in boiling dilute  $\text{H}^2\text{SO}^4$ .

4. Salts heated on charcoal with  $\text{Na}^2\text{CC}^3$  do not give a bead of metal, but a brownish incrustation.

#### GROUP II.

##### MIXTURES.

Very complicated mixtures containing Pt, Au, Sn, are not likely to occur. Supposing a solution to contain all the others, viz., As, Sb, Hg, Cu, Pb, Bi and Cd, dissolved in  $\text{HNO}^3$ .

Solution being diluted, the addition of  $\text{H}^2\text{SO}^4$  will precipitate all the Pb. In the precipitated  $\text{PbSO}^4$ , the metal can easily be detected by fusion with  $\text{NaKCO}^3$  and KCy in porcelain crucible, solution of lead in  $\text{HNO}^3$  and appropriate tests. The filtrate saturated with  $\text{H}^2\text{S}$  will give mixed sulphides, from which, by washing and warming with  $\text{Am}^2\text{S}$ , the sulphides of As and Sb (Pt, Au and Sn) would be extracted.

Addition of acid to the filtrate would precipitate  $\text{As}^2\text{S}^3$  and  $\text{Sb}^2\text{S}^3$ . These, when washed, dried and fused with  $\text{NaNO}^3$  (and  $\text{Na}^2\text{CO}^3$ ) in porcelain crucible, would give a mass which, boiled with water, leaves a residue of antimonate, soluble in HCl, to which solution tests for



Sb could be applied, and a solution containing arsenate. See under Arsenic and Arsenic Acid. The residue of black sulphides, when well washed, will, by treatment with hot  $\text{HNO}_3$ , yield up the Cu, Bi and Cd; or by boiling with dilute  $\text{H}_2\text{SO}_4$ , the CdS might be extracted, recognisable in solution by  $\text{H}_2\text{S}$ , leaving HgS, CuS and BiS. This residue, treated with  $\text{HNO}_3$ , would leave HgS, easily recognisable by reduction test with  $\text{Na}_2\text{CO}_3$ , or by solution in  $\text{HCl} + \text{HNO}_3$ , &c. Filtrate with  $\text{NH}_3$ , blue colour, copper. Filtrate evaporated to near dryness and treated with water, becomes turbid from white subsalt of bismuth.

TABLE V.—GROUP III.  
DIVISION I.—PRECIPITATE IS BLACK.  
IRON—COBALT—NICKEL.

Black precipitate easily soluble in HCl.

FERROUS SALTS.

1. Dirty white or green precipitate with HKO, turning black, and lastly brown.
2. Same with  $\text{NH}_3$ .
3. For action of  $\text{H}_2\text{S}$  and  $\text{Am}_2\text{S}$ , see Table I.
4. With  $\text{K}^4\text{Cfy}$  (Ferrocyanide) pale blue precipitate.
5. With  $\text{K}^3\text{Cfy}$  (Ferricyanide) dark blue precipitate.
6. With  $\text{KCsy}$  (Sulphocyanate) no colour, or faint red, if not pure.
7. With tannic acid, very pale blue, if pure. For making these experiments the ferrous sulphate precipitated by alcohol may be employed, and for blowpipe tests, see Table.  $\text{SO}^2$  may also be used to render the ferrous salt pure.

FERRIC SALTS.

1. Brown red precipitate with HKO, insoluble in excess.
2. Same with  $\text{NH}_3$ .
3. For action of  $\text{H}_2\text{S}$  and  $\text{Am}_2\text{S}$ , see Table I.
4. With  $\text{K}^4\text{Cfy}$  dark blue precipitate.
5. With  $\text{K}^3\text{Cfy}$  brown colour.
6. With  $\text{KCsy}$  blood red colour, destroyed by  $\text{HgCl}^2$ .
7. With tannic acid, blue black colour.

Black precipitate not easily soluble in HCl.

#### NICKEL.

1. Pale green precipitate with HKO, soluble in  $\text{NH}^3$  with blue colour.
  2. Same with  $\text{NH}^3$ , if excessively diluted or strong original solution be used, soluble in excess with blue colour.
  3. For action of  $\text{H}^2\text{S}$  and  $\text{Am}^2\text{S}$ , see Table I.
  4. With  $\text{K}^4\text{Cfy}$  a pale green precipitate, if salts are pure.
  5. With  $\text{KNO}^2$  no precipitate.
- See also Blowpipe Table.

#### COBALT.

1. Pale blue precipitate by HKO, insoluble in excess, but soluble in  $\text{NH}^3$  to brown solution. Precipitate, by standing, turns dirty green or brown.
2. Same with  $\text{NH}^3$ , if excessively diluted, soluble in excess to brown solution.
3. For action of  $\text{H}^2\text{S}$  and  $\text{Am}^2\text{S}$ , see Table I.
4. With  $\text{K}^4\text{Cfy}$  a dull green precipitate.
5. With  $\text{KNO}^2$  a yellow precipitate, especially on standing some hours.

See also Blowpipe Table. The blue colour produced in bead of borax is perhaps the best test for cobalt.

### TABLE V.—GROUP III.

#### DIVISION II.—THE PRECIPITATE IS NOT BLACK.

##### MANGANESE—CHROMIUM—ALUMINUM—ZINC.

##### MANGANESE.

1. Salts give with HKO a white precipitate, rapidly turning brown or dark coloured, especially on shaking.
2. Same with  $\text{NH}^3$ , but presence of large amount of  $\text{AmCl}$  prevents the precipitation at first.
3. For action of  $\text{H}^2\text{S}$  and  $\text{Am}^2\text{S}$ , see Table I. The buff or salmon colour of the precipitate in *pure* solutions is very characteristic. It gradually turns brown.
4. A *very* small quantity of a salt fused on platinum foil with  $\text{Na}^2\text{CO}^3$ , gives a green mass. The action is aided by the use of a small fragment of  $\text{KNO}^3$ .

See Blowpipe Table.

Salts are often pinkish.

## CHROMIUM.

1. Salts give with HKO a green or grayish precipitate, soluble in excess to fine green solution.
2. Same with  $\text{NH}^3$ , partly soluble after a time to a pink solution.
3. For action of  $\text{H}^2\text{S}$  and  $\text{Am}^2\text{S}$ , see Table I.
4. The solution in HKO gives with  $\text{AmCl}$  a green or gray precipitate, but none with little  $\text{Am}^2\text{S}$ .
5. Salts fused with nitre, with or without  $\text{Na}^2\text{CO}^3$ , give a yellow mass, soluble in water, with yellow colour. This solution gives with lead acetate a yellow precipitate, and when boiled with  $\text{HCl}$  and alcohol or  $\text{SO}^2$ , a green solution.

See Blowpipe Table.

Salts are usually green, blueish or purple.

## ALUMINUM.

1. Salts give with HKO a white precipitate, soluble in excess, often gelatinous, and not very visible for some time.
2. Same with  $\text{NH}^3$ , but insoluble in excess.
3. The solution in HKO gives a precipitate with  $\text{AmCl}$ , but not with a little  $\text{Am}^2\text{S}$ .
4. The white precipitate by  $\text{Am}^2\text{S}$  is the oxide, as with chromium, and not the sulphide.
5. Any salt or the oxide, moistened with cobalt nitrate and strongly heated, gives a blue colour.

## ZINC.

1. Salts give with HKO a white precipitate, soluble in excess.
2. Same with  $\text{NH}^3$ , soluble in excess.
3. Solution in HKO gives a precipitate with  $\text{Am}^2\text{S}$ , but not with  $\text{AmCl}$ .
4. The white precipitate formed by  $\text{Am}^2\text{S}$  is sulphide.
5. Any salt, or the oxide moistened with  $\text{Co}_2\text{NO}^3$  and heated, gives a green colour.
6. Heated on charcoal with  $\text{Na}^2\text{CO}^3$ , forms an incrustation, yellow while hot, but white when cold, giving no metallic bead.

## MIXTURE—DIVISION I.

## IRON—NICKEL—COBALT.

Black precipitate by  $\text{Am}^2\text{S}$  is well washed to remove  $\text{HNO}^3$  if present, and treated with not very strong  $\text{HCl}$ , the  $\text{FeS}$  dissolves as



ferrous salt, and may be detected by appropriate tests. The sulphides of Ni and Co can be dissolved by addition of  $\text{HNO}^3$  and  $\text{KClO}^3$ , the solution nearly neutralised with  $\text{HKO}$ ,  $\overline{\text{Ac}}$  added to distinct acid reaction, and then  $\text{HNO}^3$ , warmed and allowed to stand some hours (previous filtration may be required); a yellow precipitate indicates cobalt. Proceed further by blowpipe test. The filtrate from this yellow precipitate may be green if nickel is present, detected by appropriate tests.

#### MIXTURE—DIVISION II.

If precipitated sulphide is not black, then only the metals of this division can be present; but if dark, all of this group. In the former case the manganese may be detected by green colour produced when fused with  $\text{Na}^2\text{CO}^3$  and  $\text{KNO}^3$ ; boiled with water, a yellow solution would indicate chromium. Part of the remainder of this operation, heated on charcoal, will give incrustation if containing zinc. The rest dissolved in  $\text{HCl}$ , precipitated by  $\text{HKO}$  in excess, and solution treated with  $\text{AmCl}$  and  $\text{Am}^2\text{S}$  (see tests for Zn and Al), will indicate those metals. In the latter case, the  $\text{NiS}$  and  $\text{CoS}$ , separated by  $\text{HCl}$  as above, the filtrate oxidised by  $\text{HNO}^3$ , and treated with excess of  $\text{HKO}$ . The precipitate will contain the Fe, Mn, and probably some Cr; the solution Cr, Zn, Al, detected as above.

For detecting Fe and Cr in chromic iron ore, the substance should be very finely powdered, and fused with  $\text{KNO}^3$ . The yellow mass gives on boiling a solution of chromate, and leaves ferric oxide. It may also be fused with potassium bisulphate, when a solution of chromic and ferric oxides will be obtained. The separation of them by  $\text{HKO}$  is not complete.

#### TABLE VI.—GROUP IV.

##### BARIUM—STRONTIUM—CALCIUM—MAGNESIUM.

The oxides of first two are soluble in water; of the third, difficultly; of the fourth, almost insoluble. The carbonates are insoluble, the sulphides soluble; hence no precipitate by  $\text{Am}^2\text{S}$ , but by  $\text{Na}^2\text{CO}^3$ , or by  $\text{Am}^2\text{CO}^3$ , especially on boiling (magnesium).

##### BARIUM.

- + 1. Salts give an immediate precipitate with  $\text{CaSO}^4$ , insoluble in acids.



2. Salts, in moderately strong solutions, give a precipitate with sodium hyposulphite ( $\text{Na}^2\text{S}^2\text{O}^3$ ), especially on shaking.

3. Dry salts, after slightly moistening and warming, communicate a greenish-yellow colour to the flame of alcohol. The same colour is observed when the salt, even if insoluble, is heated on platinum wire. Glycerine is said to assist the formation of the colour.

4. Salts are precipitated by potassium chromate ( $\text{K}^2\text{CrO}^4$ ).

#### STRONTIUM.

1. Salts are precipitated after a time (five or ten minutes) by  $\text{CaSO}^4$ .

2. Salts are not precipitated by  $\text{Na}^2\text{S}^2\text{O}^3$ .

3. Salts communicate fine crimson colour to flame of alcohol or blowpipe.

4. Salts are not precipitated by  $\text{K}^2\text{CrO}^4$ .

#### CALCIUM.

1. Salts are not precipitated by  $\text{CaSO}^4$ , but if very dilute  $\text{H}^2\text{SO}^4$  be used instead of  $\text{CaSO}^4$ , so dilute as not to produce a precipitate, and then alcohol added, a precipitate will be formed,  $\text{CaSO}^4$  being insoluble in dilute alcohol.

2. Salts give no precipitate with  $\text{Na}^2\text{S}^2\text{O}^3$ .

3. Salts give an orange red colour to flame of alcohol or blowpipe.

4. Salts are not precipitated by  $\text{K}^2\text{CrO}^4$ .

5. Salts are precipitated by ammonium oxalate, or oxalic acid and ammonia. The precipitate is insoluble in acetic acid, soluble in  $\text{HCl}$  without effervescence, but if dried and heated, with effervescence.

#### MAGNESIUM.

1. Salts not precipitated by  $\text{CaSO}^4$ .

2. Salts not precipitated by  $\text{Na}^2\text{S}^2\text{O}^3$ .

3. Salts give no colour to alcohol flame.

4. Salts not precipitated by  $\text{K}^2\text{CrO}^4$ .

5. Salts not precipitated by  $\text{Am}\overline{\text{Ox}}$ , but by  $\text{Na}^2\text{HPO}^4$ , with addition of  $\text{Am}^2\text{CO}^3$ ; precipitate sometimes crystalline, and soluble in acetic acid; also by ammonium arsenate.

6. Salts precipitated by  $\text{NH}^3$  partly, the precipitate soluble in  $\text{AmCl}$ . Hence use of  $\text{AmCl}$  in applying the test for Group III., to avoid precipitation of  $\text{MgO}$ .

## GROUP IV.

Mixture—Ba, Sr, Ca, Mg.

The precipitated carbonates dissolved in as little HCl as possible;  $\text{NH}_4\text{Cl}$  added in excess, and then ammonium carbonate, and boiled; the precipitate filtered and washed.

The filtrate will contain the Mg, detected, as already described, by  $\text{Na}_2\text{HPO}_4$  or  $\text{Am}^2\text{HAsO}_4$ .

The precipitate dissolved in as little HCl as possible, evaporated to dryness, re-dissolved.

One portion tested with  $\text{CaSO}_4$ , an immediate precipitate indicates Ba.

Another portion treated with  $\text{K}_2\text{CrO}_4$  till no more precipitate is formed, filtered, the precipitate is  $\text{BaCrO}_4$ . The filtrate may contain strontium, detected by  $\text{CaSO}_4$ , &c.

Another portion precipitated fully by  $\text{K}_2\text{SO}_4$ , or  $\text{Na}_2\text{SO}_4$ , filtered, the filtrate will contain Ca, but only in small quantity, detected by  $\text{Ox}$  and  $\text{NH}_3$ . The insolubility of  $\text{BaCl}_2$  in alcohol may also be taken advantage of in the above separation.

When a mixture of these salts is heated on platinum foil, such as the sulphates, more especially with glycerine, the orange and red colour of the Ca and Sr will first be apparent, and the green of Ba later.

The precipitate obtained as above, by *boiling* with  $\text{Am}^2\text{CO}_3$ , may contain some magnesia, which, however, will not interfere with the subsequent tests. Perhaps it may be better only to warm for some time; the object of boiling is to throw down all the  $\text{CaCO}_3$ , some of which might remain in solution.

## TABLE VII.—GROUP V.

POTASSIUM — SODIUM — AMMONIUM.

The hydrates (of Am unknown), carbonates, phosphates, sulphides, and all salts, with few exceptions, are easily soluble in water. The hydrates of K and Na strongly and persistently alkaline, as well as many salts. The hydrate (?) of Am evanescently alkaline; also the carbonates. Hydrate of Li persistently alkaline; also the carbonate, but the latter salt not so easily soluble, this metal forming a connecting link between Groups IV. and V.

## POTASSIUM.

1. Salts in rather strong solutions give a crystalline precipitate with  $\overline{\text{Tar}}$ , if well shaken. If the hydrate or carbonate be employed, care must be taken to render the solution fully acid. Hydric sodium tartrate,  $\text{HNaC}^4\text{H}^4\text{O}^6$  (sodium bitartrate), is in some cases preferable to  $\overline{\text{T}}$ .

2. Salts in not too dilute solutions, acidulated with  $\text{HCl}$ , give a yellow precipitate with  $\text{PtCl}^4$ .  $\text{PtCl}^4 + 2\text{KCl}$ . To avoid using much  $\text{PtCl}^4$ , the experiment may be made with a drop or two of the solution on a glass plate.

3.\* Salts burnt with alcohol, or heated on platinum wire, give a violet colour, especially visible when viewed through blue glass. Fluosilicic and picric acids also give precipitates with strong solutions of potassium salts.

## SODIUM.

1. Salts give no precipitate with  $\text{PtCl}^4$ , the compound produced being easily soluble in  $\text{H}^2\text{O}$ .

2. Salts give no precipitate with  $\overline{\text{T}}$ , the tartrates being soluble in  $\text{H}^2\text{O}$ ; the same may be said with regard to fluosilicic and picric acids.

8. All salts of sodium, when burnt with alcohol, or heated in blowpipe flame, give a yellow colour, which vanishes when viewed through a blue glass. The most minute trace of sodium salt may thus be detected, more especially by the aid of the spectroscope; the persistent or flashing yellow line is a complete proof. Potassium antimonate has been proposed as a test for sodium salts; it is not necessary.

## AMMONIUM.

1. Salts of this hypothetical metal give, in not very dilute solutions, acidulated with  $\text{HCl}$ , a yellow precipitate of  $\text{PtCl}^4\text{Am}^2\text{Cl}^2$ .

2. Salts of ammonium give no precipitate with tartaric acid or hydric sodium tartrate, unless *very* concentrated.

3. Salts of ammonium give no colour to the flame of alcohol.

4. Salts of ammonium are volatilised or decomposed by heat, forming in many cases an amorphous sublimate.

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\* In making these alcohol flame tests, observe the directions given in previous note.



5. Salts of ammonium give off  $\text{NH}^3$  when drenched with solution of  $\text{HKO}$ , or heated with lime. The  $\text{NH}^3$  detected by odour, and by giving white fumes with  $\text{HCl}$ .

6. All salts of ammonium, even in most minute quantity, will give a yellow or brown colour, or precipitate with Nessler's Solution.  $\text{HgI}^2\text{K}^2\text{I}^2 + \text{HNaO}$ . The most delicate test for  $\text{NH}^3$  or its combinations if in solution. The precipitate formed is iodide of mercuric ammonium,  $\text{NHg}^2\text{I}$ , or some analogous compound.

#### LITHIUM.

1. The carbonate being only partly soluble in water, a precipitate is formed in concentrated solutions by sodium carbonate, especially on warming; none in dilute when cold.

2. The same is the case with the phosphate in concentrated solutions, lithium standing between the Groups IV. and V.

3. The splendid crimson colour shewn by salts of lithium when heated on platinum wire is probably the best test; also the marked red line, as seen by the spectroscope.

#### GROUP V.

##### IN MIXTURE.

In a mixture, the metals of Groups I. and II. may be separated by  $\text{H}^2\text{S}$ , and of III. by  $\text{Am}^2\text{S}$ ; from the filtrate Ba Sr and Ca by  $\text{Am}^2\text{CO}^3$ , from the filtrate, the Mg by lime or baryta water, warming, filtering. The filtrate, treated with  $\text{AmCO}^3$ , warmed and filtered, will contain K, Na, and of course salts of Am. If evaporated to dryness and heated, then the latter will be expelled and only K and Na remain, detected by alcohol flame and  $\text{PtCl}^4$ . If presence of a salt of Am is suspected, it must be looked for in dry original salt by the  $\text{HKO}$  test. If none of the first four groups are present, the salts may be examined at once for Am, heated, and the residue tested for K and Na. Lithium detected by crimson colour in blowpipe flame. The magnesium may also be separated by means of ammonium phosphate or arsenate (Attfield). These reagents have not been mentioned in preceding pages, but can be easily prepared by neutralising phosphoric or arsenic acids by  $\text{Am}^2\text{CO}^3$ . After the precipitation of Mg, the filtrate, evaporated to dryness and strongly heated, may contain only K and Na, with of course phosphoric or arsenic acids.



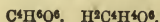
## ACIDS, OR SALT RADICALS.

The detection of these is not so satisfactory as that of the bases, owing to the fact that the groups run into one another, while with the bases they are perfectly distinct. The student must distinguish between decided blackening on platinum foil, or preferably in tube, as shewn by tartrates, &c., and the darkening or browning exhibited by oxalates, ferro and ferricyanides. If any doubt arises, or if a solution has to be employed, then the Supplementary Table may be used. A fresh portion of original solution, made neutral by  $\text{NH}^3$ , should be used for columns I. and II.; all excess of  $\text{NH}^3$  should be got rid of by boiling when at column III.

In examining for acids, the action of heat on the salt, either on platinum foil or in a tube, should be tried, and the heating continued even if the salt fuses. Sodium acetate will melt in water of crystallisation, and might be relegated to inorganic class; if still further heated, it will blacken. The next operation should be that described in Group IV., viz., deflagration on charcoal; this should have been printed Group II. In using Group Test II., the solution should be neutral, or nearly so; in Group III. the solution may be either neutral or acid; if acid, preferably by  $\text{HNO}^3$ .

It may happen that the substance contains no metallic base unless H, and is an acid. The acidity is easily recognised by reddening action on blue litmus paper; but there are some acid salts and metallic salts which redden blue litmus. If a substance in solution is supposed to be an acid, *i.e.*, a free acid, it may be neutralised by HKO; if an acidly reacting metallic salt, a precipitate will be formed at once. If, on the other hand, it is an acid, the addition of HKO will produce no precipitate, unless the salt so formed should be insoluble, or difficultly soluble in  $\text{H}^2\text{O}$ . The solution thus obtained may be evaporated to dryness, and the residue tested according to usual plan.

## TARTARIC ACID.

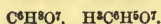


1. The acid, and all salts, blacken when heated, evolving a peculiar smell, something like that of burnt sugar. Characteristic test.
2. The acid added in excess to HKO or to  $\text{KC}^2\text{H}^3\text{O}^2$  (potassium acetate), and well shaken, gives a crystalline precipitate of  $\text{HKC}^4\text{H}^4\text{O}^6$  (potassium bitartrate). The same result will occur on using a neutral salt with potassium acetate if a little HCl be added.

3. Acid gives with *large* excess of lime water  $\text{H}^2\text{CaO}^2$ ; a white precipitate in the cold. Neutral salts give the same with  $\text{CaCl}^2$ . The precipitate is soluble in large excess of HKO. It is easily soluble in  $\bar{\text{Ac}}$  when freshly prepared, but when precipitated from ammoniacal solutions it is apt to become crystalline and insoluble in  $\bar{\text{Ac}}$ , and might be mistaken for calcium oxalate. They are both however soluble in HCl; the oxalate is precipitated immediately by  $\text{NH}^3$ , the tartrate is not.

4. Acids and salts are blackened rapidly by heating with  $\text{H}^2\text{SO}^4$ , evolving a mixture of gases, among which carbon monoxide may sometimes be detected, by its burning with a blue flame.

#### CITRIC ACID.



1. The acid and its salts blacken when heated, and evolve vapours of an acid character.

2.  $\text{CaCl}^2$  produces in neutral salts a precipitate only after boiling.

3.  $\text{H}^2\text{CaO}^2$  acts in the same way; reagent must be used in large excess.

4. The acid and salts, when heated with  $\text{H}^2\text{SO}^4$ , form a yellow solution, blackening only after a considerable time.

5. The acid, even in excess, gives no precipitate with HKO.

#### URIC ACID.

1. The acid and salts blacken when heated.

2. The acid is insoluble, the salts generally difficultly soluble in water even when boiling (the ammonium salt an exception), but easily soluble in hot HKO, from which solution the acid may be reprecipitated by addition of HCl.

8. The acid or salts, treated with a little  $\text{HNO}^3$  in a capsule, evolve red fumes. On evaporating to perfect dryness, avoiding too much heat, so as not to char, the residue becomes bright red, turning to fine purple on addition of HKO.

#### TANNIC ACID.



There are several varieties of this acid, most of them giving a blue-black colour or precipitate with  $\text{Fe}^2\text{Cl}^6$ , but some a green. Most of them are precipitated by gelatine. They are generally considered as

glucosides, *i. e.*, are resolved by ferment or by dilute acids into X and glucose. Some doubt has lately been thrown on this idea. The only variety which need be here considered is that obtained from oak-galls.

1. Pure ferrous salts produce a blue colour, turning black on addition of potassium acetate.

2. Ferric salts produce a blue-black colour or precipitate (ink).

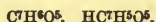
3. Solution of gelatine or albumen produces a precipitate.

4. Hot  $\text{H}^2\text{SO}^4$  rapidly produces blackening.

5. In presence of free alkalis, soon becomes dark-coloured from absorption of oxygen.

6. Solution of acid made faintly alkaline by  $\text{NaHCO}^3$  or any other alkaline salt, turns green after standing for some hours.

#### GALLIC ACID.



1. Pure ferrous salt produces a blue colour; dark blue on addition of potassium acetate.

2. Ferric salts produce a blue-black colour or precipitate (old ink).

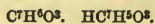
3. Solution of gelatine or albumen produces no precipitate.

4. Hot  $\text{H}^2\text{SO}^4$  rapidly produces blackening.

5. In presence of alkalis soon becomes dark-coloured from absorption of oxygen.

6. Solution of acid made faintly alkaline by  $\text{NaHCO}^3$ , or any other alkaline salt, becomes dark-green after a few hours.

#### BENZOIC ACID.



1. Salts blacken when heated, except that of  $\text{NH}^4$ .

2. Solutions of salts give with stronger acids a precipitate of benzoic acid, which, when separated and dried, is easily volatilised, giving pungent irritating vapours, condensing to fine acicular crystals, with characteristic smell.

3. Salts give a pale brown precipitate with  $\text{Fe}^2\text{Cl}^6$ .

4. Acid itself, when boiled with water, melts; the supernatant solution deposits crystals on cooling; the steam evolved has peculiar smell of benzoic acid.



## ACETIC ACID.



1. Salts are blackened more or less, especially when heated in a close tube.  $\text{NH}^4\text{C}^2\text{H}^3\text{O}^2$  will not blacken like many other  $\text{NH}^4$  salts.

2.  $\text{Fe}^2\text{Cl}^6$  produces a red colour, not altered by  $\text{HgCl}^2$ , but destroyed by  $\text{HCl}$ , or by boiling under formation of brown-red precipitate. Hence distinguished from meconic and sulphocyanic acids. See Supplementary Table.

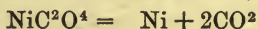
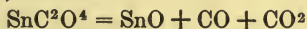
3. Dry salts, heated with moderately strong  $\text{H}^2\text{SO}^4$ , evolve smell of vinegar.

4. Dry salts, heated with  $\text{H}^2\text{SO}^4$  and pure alcohol, evolve sweet smell of acetic ether.  $\text{C}^2\text{H}^5\text{C}^2\text{H}^3\text{O}^2$ .

5. Dry salt, heated with  $\text{As}^2\text{O}^3$ , evolves horribly smelling alkarsin.  $\text{C}^4\text{H}^6\text{As}$ . Care must be taken in making this experiment, the alkarsin being so exceedingly poisonous. The addition of  $\text{SnCl}^2$  (dry) will cause the formation of the chloride, which has, if possible, a worse smell. Ammonium acetate will volatilise without any but the slightest blackening.

## OXALIC ACID.

1. The acid itself volatilises on heating without blackening; the salts darken very slightly—that of  $\text{NH}^4$  scarcely at all; the residue, when any, dissolves in acids with effervescence, chiefly seen in the oxalates of the earths and alkalies. Those of the heavier metals leave as residue either the metallic oxide,  $\text{FeC}^2\text{O}^4$ ,  $\text{SnC}^2\text{O}^4$ , or the metal itself  $\text{NiC}^2\text{O}^4$ ,  $\text{CoC}^2\text{O}^4$ .



2. The precipitate, with salts of calcium, is soluble in  $\text{HCl}$  without effervescence, insoluble in  $\overline{\text{Ac}}$ ; after heating (when dried), soluble in both with effervescence.  $\text{CaC}^2\text{O}^4 = \text{CaCO}^3 + \text{CO}$ . The  $\text{CO}$  is driven off.

3. Acid or salts heated with  $\text{H}^2\text{SO}^4$  evolve gas ( $\text{CO}^2 + \text{CO}$ ), which burns with a blue flame. If passed into lime water produces a precipitate of calcium carbonate.  $\text{K}^2\text{C}^2\text{O}^4 + \text{H}^2\text{SO}^4 = \text{K}^2\text{SO}^4 + \text{CO}^2 + \text{CO} + \text{H}^2\text{O}$ .

## FERROCYANIC.



1. Salts when heated turn more or less brown.

2. Heated with moderately strong  $\text{H}^2\text{SO}^4$ , evolve smell of  $\text{HCy}$ .



3. Heated with excess of strong  $\text{H}^2\text{SO}^4$ , evolve  $\text{CO}$ , a gas burning with blue flame.

4. Give pale blue precipitate with ferrous salt.

5. Give dark blue precipitate with ferric salt.

6. Give granular precipitate with  $\text{BaCl}^2$ ,  $\text{Ba}^2\text{Cfy}$ , or compound with  $\text{K}^4\text{Cfy}$ , insoluble in  $\text{Ac}$ .

#### FERRICYANIC.

$\text{H}^2\text{Cfy}$ .

1. Salts turn brown more or less on heating.

2. Give dark blue precipitate with ferrous salt.

3. Give brown colour with ferric salt.

#### SULPHOCYANIC.

$\text{HCys}$ .

1. Some salts blacken only very slightly when heated.

2. Give no colour with pure ferrous salt.

3. Give blood-red colour with ferric salt; colour destroyed by  $\text{HgCl}^2$ , not by boiling.

4. Salts give white or grayish precipitate with mercuric nitrate, which, when washed, dried and inflamed, swells up enormously.

#### SULPHUROUS ACID.

$\text{H}^2\text{SO}^3$  or  $\text{SO}^2$ .

1.  $\text{H}^2\text{SO}^3$  in solution, or  $\text{SO}^2$  as gas, is recognised by smell, or by action on paper soaked in solution of starch and iodic acid, producing a blue colour from free  $\text{I}$  acting on starch.  $\text{I}^2\text{O}^5 + 5\text{SO}^2 = \text{I} + 5\text{SO}^3$ , leaving water out of consideration, or by reducing action on acidulated solutions of  $\text{K}^2\text{CrO}^4$  or  $\text{KMnO}^4$ .

2.  $\text{BaCl}^2$  gives a precipitate of  $\text{BaSO}^3$  with sulphites soluble in  $\text{HCl}$ . Generally the commercial sulphites contain some sulphates, and the precipitate is not entirely soluble.

3. The dry sulphites evolve a gas smelling of burning sulphur when treated with stronger acids; but as the  $\text{SO}^2$  is very soluble in  $\text{H}^2\text{O}$ , this experiment will not succeed with solutions unless very concentrated; it is better to employ the dry salt.

4. Also detected by evolving  $\text{H}^2\text{S}$ , when warmed with zinc and  $\text{HCl}$ , the  $\text{H}^2\text{S}$  recognised by action on lead paper.  $\text{SO}^2 + \text{Zn}^2 + \text{H}^2\text{Cl}^2 = \text{H}^2\text{S} + \text{H}^4\text{O}^2 + \text{Zn}^2\text{Cl}^4$ .

## SULPHURIC ACID.



1. When concentrated, may be recognised by its blackening action on wood or paper; even when dilute, the smear or writing effected therewith will become black on gently heating by the charring. Lignine (wood) or cellulose (paper) may be represented as being carbon + water; the sulphuric acid removes the water.

2. Free or in combination by giving an immediate precipitate with soluble salts of Ba; the precipitate is insoluble in all ordinary acids.  $\text{BaCl}^2 + \text{H}^2\text{SO}^4 = \text{H}^2\text{Cl}^2 + \text{BaSO}^4.$

3. All sulphates, when *well* fused on charcoal with  $\text{Na}^2\text{CO}^3$ , give a hepar, generally of reddish colour; this fused mass, when placed on a silver coin and moistened with water, gives a brown stain; if treated with a little dilute  $\text{H}^2\text{SO}^4$ , evolves  $\text{H}^2\text{S}$ . The re-actions may be represented thus:  $\text{Na}^2\text{SO}^4 + \text{C}^4 = \text{Na}^2\text{S} + \text{C}^4\text{O}^4.$  The alkalic sulphides, especially if containing excess of S, act on Ag, producing  $\text{Ag}^2\text{S}$ . The action of acid is:  $\text{Na}^2\text{S} + \text{H}^2\text{SO}^4 = \text{H}^2\text{S} + \text{NaSO}^4.$

4. The hepar dissolved in water, gives with sodium nitroprusside a fine purple colour; the reaction cannot be represented.

## PHOSPHORIC ACID.



1. Common phosphates produce, in concentrated solutions of magnesium salts, a slight precipitate; in presence of ammonia and ammoniacal salts, a precipitate more or less crystalline (see Plates); easily soluble in excess of Ac. See Urine.

2. Phosphates ( $\text{MgAm PO}^4$ ) produce a white precipitate with salts of lead, which, when filtered, washed, dried and heated on charcoal or on platinum wire, forms an opaque bead, which, on cooling, exhibits crystalline structure.

3. A small quantity of phosphate, mixed with excess of ammonium molybdate, gives yellow colour, and yellow precipitate on warming. There must always be an excess of molybdate and free acid.

4. Phosphates give, with  $\text{AgNO}^3$ , a yellow precipitate, easily soluble in nitric but not easily in acetic acid; easily soluble in  $\text{NH}^3$ .

5. This latter test may be applied to some insoluble phosphates; addition of  $\text{AgNO}^3$  will often produce a yellow colour; if the earthy phosphate be dissolved in the least trace of  $\text{HNO}^3$ , then  $\text{AgNO}^3$  added and the faintest trace of  $\text{NH}^3$ , the acid may usually be

detected by the yellow colour produced. The fact of the yellow precipitate formed by  $\text{AgNO}_3$  being insoluble in  $\overline{\text{Ac}}$ , distinguishes phosphoric from arsenious acid.

6. If a not very strongly acid solution of a phosphate be mixed with sodium acetate and ferric chloride, a yellowish brown precipitate will be formed, and, on boiling, the whole of the acid will be thrown down in combination with  $\text{Fe}_2\text{O}_3$ .

#### BORACIC ACID.

$\text{B}_2\text{O}_3$  or  $\text{H}^3\text{BO}_3$ .

1. A strong solution of any salt, when acidulated with  $\text{H}^2\text{SO}_4$  or  $\text{HCl}$ , deposits, on cooling, crystalline scales of boracic (boric) acid.

2. If alcohol be added to the above mixture, warmed and kindled, it will burn with a bright green flame.

3. A solution of borate or of acid, mixed with  $\text{HCl}$ , will turn turmeric paper red-brown, especially after drying. The colour is changed by  $\text{HKO}$  to green.

4. Borates heated on platinum wire with glycerine, all give a more or less distinct green colour.

#### CARBONIC ACID.

$\text{CO}_2$  or  $\text{H}^2\text{CO}_3$ .

*d* 1. The gas is readily detected by giving a white precipitate with lime water, when shaken. The gas, if evolved from a salt by action of stronger acid, may be poured out of the tube in which the experiment is made, into another containing some lime water.

2. All carbonates effervesce on addition of stronger acid; gas evolved has little or no smell.

3. Soluble carbonates give precipitates with  $\text{CaCl}_2$  and  $\text{BaCl}_2$ ; soluble in acids with effervescence, after being washed.

#### SILICIC.

$\text{SiO}_2$  or  $\text{H}^4\text{SiO}_4$ .

1. Soluble silicates, when in concentrated solutions, give a gelatinous precipitate with  $\text{HCl}$ , no precipitate in dilute solutions; when evaporated to dryness and treated with  $\text{H}_2\text{O}$ , an insoluble residue is left.

2. Fused with  $\text{Na}^2\text{CO}_3$  on platinum wire, give, under effervescence, a bead which is transparent while hot.



3. Silicates fused on platinum wire with borax or phosphor salt, give a skeleton of  $\text{SiO}_2$ .

4. With ammonium molybdate, a yellow precipitate on warming like phosphates.

#### HYDROFLUORIC.

$\text{HF}$ .

1. The most common occurring substances containing fluorine are cryolite, apatite, bones, and especially enamel.

2. Best detected by coating a plate of glass with thin layer of wax, removing some portion of this by pointed instrument, and exposing the plate to the vapours of  $\text{HF}$ , evolved when the compound is gently warmed in a platinum or leaden dish. The glass will be seen to be etched on cleaning with oil of turpentine.

3. Fluorides heated with glass or quartz and  $\text{H}^2\text{SO}^4$ , evolve a gas which, when passed into  $\text{H}^2\text{O}$ , deposits a flocculent precipitate of  $\text{SiO}_2$ , and gives an acid solution of fluosilicic acid  $\text{H}^2\text{SiF}^6$ .

#### CHROMIC ACID,

$\text{CrO}_3$ .

1. Salts are yellow, red or brown.

2. Reduced to green solution of chromic oxide by heating with  $\text{HCl}$  and alcohol or  $\text{SO}^2$ .

3. Give yellow precipitate with salts of lead, the precipitate soluble in large excess of  $\text{HKO}$ .

4. Give dark crimson precipitate with  $\text{AgNO}_3$ .

5. Dry salts mixed with dry  $\text{NaCl}$  and  $\text{H}^2\text{SO}^4$ , and heated, give dark red-brown vapours. These act upon solution of  $\text{NH}_3$ , or paper soaked in  $\text{NH}_3$ , producing yellow colour; hence distinguished from bromine vapours. This coloured vapour is not produced by bromides or iodides, hence means of detecting chlorides; but the action of  $\text{H}^2\text{SO}^4$  on the former must be remembered, viz., producing both  $\text{Br}$  and  $\text{HBr}$ .

#### ARSENIC ACID.

$\text{As}_2\text{O}_3$  or  $\text{H}^3\text{AsO}_4$ .

1. No precipitate by  $\text{HKO}$  or  $\text{NH}_3$ .

2. Precipitate by  $\text{H}^2\text{S}$  of  $\text{As}_2\text{S}_5$ , only after a time. Solution of  $\text{H}^2\text{S}$  seldom produces a precipitate even in an acid solution, unless used in large excess and warmed. The precipitate corresponds in most respects with that formed from arsenious acid, but appears to be  $\text{As}_2\text{S}^3 + \text{S}_2$ .



3. Arsenates, mixed with  $\text{MgSO}^4$  and  $\text{NH}^3$ , give a white, more or less crystalline precipitate, soluble in acetic acid, corresponding to the similar salt formed by phosphoric acid.  $\text{MgAm}$ ,  $\text{AsO}^4$ .

4.  $\text{AgNO}^3$  produces a red-brown precipitate, rather difficultly soluble in acetic acid; the arsenite being easily soluble, it is easy to detect in this manner a mixture of arsenate with arsenite, as frequently occurs in old *Liquor Arsenicalis*.

5. The remaining reactions are the same as with arsenious acid.

### ARSENIOUS.

See under Arsenic.

### HYDROCHLORIC ACID.

$\text{HCl}$ .

1. Salts warmed with  $\text{H}^2\text{SO}^4$  evolve a pungent gas,  $\text{HCl}$ , producing fumes in air, more especially with a rod moistened with  $\text{NH}^3$  held over the capsule in which the experiment is made.  $\text{HCl} + \text{NH}^3 = \text{NH}^4\text{Cl}$ .

2. Salts warmed with  $\text{H}^2\text{SO}^4$  and  $\text{MnO}^2$ , evolve  $\text{Cl}$ , recognised by odour and bleaching power.  $\text{Na}^2\text{Cl}^2 + \text{MnO}^2 + 2\text{H}^2\text{SO}^4 = \text{Cl}^2 + \text{Na}^2\text{SO}^4 + \text{MnSO}^4 + \text{H}^4\text{O}^2$ .

3. Salts heated with  $\text{H}^2\text{SO}^4$  and dry  $\text{K}^2\text{CrO}^4$ , evolve dark red fumes of chlorochromic acid,  $\text{CrO}^2\text{Cl}^2$ .

4. With  $\text{AgNO}^3$  give on shaking a white curdy precipitate, insoluble in dilute acids, soluble in  $\text{NH}^3$ ,  $\text{KCy}$  and  $\text{Na}^2\text{S}^2\text{O}^3$ , blackened by exposure to light.

### HYDROBROMIC ACID.

$\text{HBr}$ .

1. Bromides heated with  $\text{H}^2\text{SO}^4$  evolve white fumes of  $\text{HBr}$  and yellow  $\text{Br}$ , recognised by smell and bleaching power. With addition of  $\text{MnO}^2$ , only  $\text{Br}$  will be evolved.

2. Solutions of salts treated with  $\text{Cl}$  or  $\text{Cl}$  water, acquire a yellow colour, removed by shaking with ether, which dissolves the free bromine.

3. Free bromine colours starch yellow or reddish, best shewn by placing a paper soaked in starch paste with some dry starched sprinkled on it, over the mouth of the tube or glass in which the bromine is evolved. The re-action is not very striking.

4. Bromides give with  $\text{AgNO}_3$  a pale yellow precipitate, soluble in large excess of  $\text{NH}_3$ , especially on warming; soluble in  $\text{KCy}$  and  $\text{Na}_2\text{S}_2\text{O}_4$ .

### HYDRIODIC ACID.

#### HI.

1.  $\text{AgNO}_3$  produces a pale yellow precipitate of  $\text{AgI}$ , insoluble in  $\text{NH}_3$ , but becoming lighter in colour; soluble in  $\text{KCy}$  or  $\text{Na}_2\text{S}_2\text{O}_4$ , insoluble in  $\text{HNO}_3$ . In making this test, enough  $\text{AgNO}_3$  must be added to decompose all the iodide, otherwise a brown colour will be produced from liberated iodine.

2.  $\text{HNO}_3$ ,  $\text{Cl}$  water or  $\text{Br}$  water, added to solutions of iodides, produce a yellow or brown colour if added in small, a brown precipitate if added in larger quantity. An excess of  $\text{Cl}$  or  $\text{Br}$  will dissolve the precipitate to a colourless solution, hence reagent mentioned in test 5 is preferable where small quantities have to be detected.

3. Taking the pale yellow solution thus obtained, or diluting the brown solution till of a lighter colour, the presence of free iodine ( $\text{KI} + \text{Cl} = \text{KCl} + \text{I}$ ) may be proved by—

(a) Addition of starch paste, blue colour.

(b) Shaking with a few drops of carbon disulphide  $\text{CS}_2$ , or chloroform  $\text{CHCl}_3$ , when the liquid becomes pink or dark if much iodine be free, on standing the  $\text{CS}_2$  or  $\text{CHCl}_3$  sinks to the bottom, with a pink or dark colour, from their power of removing the free iodine. Benzol or benzoline may be used instead of the above, but it will, of course, rise to the top.

4. Iodides, *i.e.*, where the iodine is combined with a metal, have of course no action on starch; the iodine must be set free. A very delicate method of applying the test is to add to the very dilute solution of iodide, starch paste a few drops of  $\text{HCl}$ , then one drop of potassium chromate  $\text{K}_2\text{CrO}_4$ ; on standing, the blue colour will be produced. By this means the iodine may be detected directly in some few Canadian mineral waters.

5. Instead of  $\text{Cl}$  or  $\text{Br}$ , a preferable reagent is  $\text{H}_2\text{SO}_4$ , into which has been passed the gas  $\text{N}_2\text{O}_3$ , arising from the action of  $\text{HNO}_3$  on starch or  $\text{As}_2\text{O}_3$ . This does not destroy the blue colour when used in excess, and does not set free bromine from bromides.

6. Lead acetate produces a yellow precipitate, soluble in very large excess of boiling water.

7. Iodides heated in dry state with  $\text{H}^2\text{SO}^4$ , evolve violet fumes of I, with little or no HI.

Platinic and palladous chlorides are also excellent tests for iodine, producing brown colour and precipitate. By  $\text{PdCl}^4$  the I can be detected directly in some Canadian waters. The reagent, however, is not generally procurable.

### HYDROCYANIC.

#### HCy.

1. In all experiments with this acid special care should be taken, it being so exceedingly poisonous. The smell of the acid when once noticed can never be mistaken; it is said to resemble that of oil of bitter almonds. This oil in its crude state contains HCy; the smell of the pure oil is quite different from that of HCy.

2. All cyanides (excepting mercuric) evolve HCy when treated with dilute  $\text{H}^2\text{SO}^4$ . For accurate methods of detection, see chapter on Poisons.

3. A solution of HCy or a cyanide, gives with  $\text{AgNO}^3$  a white curdy (on shaking) precipitate, soluble in  $\text{NH}^3$ , not soluble in  $\text{HNO}^3$ , unless by boiling a long time with concentrated acid. The precipitate is soluble in  $\text{KCy}$ ,  $\text{Na}^2\text{S}^2\text{O}^4$ , &c.

4. The precipitate so obtained, collected on filter, washed, dried, introduced into a narrow quill tube and heated, gives off a gas which may be made to burn at the mouth of the tube with a violet flame.

5. Into another narrow quill tube a small fragment of iodine is introduced; only a fraction of a grain; on the top of that is placed some of the dried  $\text{AgCy}$ ; *very* gentle heat is then applied, when a ring or deposit of fine acicular crystals of  $\text{CyI}$  will be found filling up the tube.

6. HCy, or a soluble cyanide mixed with an old solution of  $\text{FeSO}^4$ , or what is the same, pure  $\text{FeSO}^4$  with a few drops of ferric salt ( $\text{Fe}^2\text{Cl}^6$ ) mixed with  $\text{HKO}$ , gives a dirty green or blue, or brown precipitate. On addition of  $\text{HCl}$ , a fine blue colour is produced. When the quantity of HCy is very small, only a green colour may be visible, but on standing a blue sediment will settle.

7. HCy mixed with a few drops of  $\text{Am}^2\text{S}$  in a capsule, and gently warmed until all smell has been dispelled, or until the mixture has been evaporated to dryness, gives with  $\text{Fe}^2\text{Cl}^6$  a blood-red colour.

Care must be taken that the whole excess of  $\text{Am}^2\text{S}$  is driven off, and also that the residue is not heated more than to dryness.

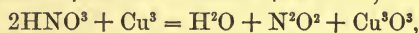
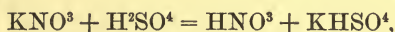
For other methods of applying this test, see under Hydrocyanic Acid in chapter on Poisons.

### NITRIC ACID.



+

1. Salts deflagrate on charcoal when heated.
2. Give no precipitate, if pure, with  $\text{BaCl}^2$  or  $\text{AgNO}^3$ ; in using  $\text{BaCl}^2$  the solution must be dilute, otherwise a precipitate may occur from formation of  $\text{Ba}^2\text{NO}^3$ , which is not very soluble in water.
3. Heated with  $\text{H}^2\text{SO}^4$  and metallic copper, the salts evolve red fumes from evolution of  $\text{N}^2\text{O}^2$ , and subsequent change of this into  $\text{N}^2\text{O}^3$  or  $\text{N}^2\text{O}^4$ .



which dissolves in the  $\text{H}^2\text{SO}^4$ .

4. Acid or salt with  $\text{H}^2\text{SO}^4$ , warmed with few drops of  $\overline{\text{Ind}}$  (sulphindigotic acid), destroys the blue colour.

### CHLORIC ACID.



1. Salts deflagrate violently on charcoal.
2. Give no precipitate, if pure, with  $\text{BaCl}^2$  or  $\text{AgNO}^3$ ; if strongly heated to perfect fusion and effervescence, the residue when dissolved gives a precipitate with  $\text{AgNO}^3$ .
3. Treated with  $\text{H}^2\text{SO}^4$  turn yellow when warmed, evolving a gas which is very explosive. Care must be taken in making this experiment.
4. Same action on  $\overline{\text{Ind}}$  as salts of  $\text{HNO}^3$ ; the salt should be used in solution, and not dry, to avoid chance of explosion.
5. Salts acted on by  $\text{HCl}$  evolve a mixture of  $\text{Cl}$  and  $\text{Cl}^2\text{O}^4$ , easily recognisable by bleaching power.

### PERCHLORIC.



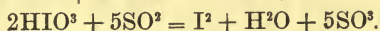
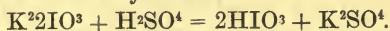
1. Salts deflagrate on charcoal like chlorates.
2. No precipitate with  $\text{AgNO}^3$  until after strong heating.
3. Salts do not turn yellow with  $\text{H}^2\text{SO}^4$ , or evolve an explosive gas.
4. They are not acted on by  $\text{HCl}$ .



## IODIC.



1. Salts deflagrate on charcoal, giving off violet fumes.
2. Give precipitate with  $\text{AgNO}^3$  and  $\text{BaCl}^2$ .
3. Heated with  $\text{H}^2\text{SO}^4$ , evolve violet fumes.
4. Treated with starch paste and  $\text{SO}^2$ , give blue colour. In case of iodates a little  $\text{H SO}^4$  may be added.



## BROMIC.



1. Salts deflagrate on charcoal.
2. Heated alone, or with  $\text{H}^2\text{SO}^4$ , evolve yellow vapours of bromine.
3. Solutions reduced by  $\text{SO}^2$ , with production of yellow colour; removed by ether.

## MINERAL AND POTABLE WATERS.

The analysis of these belongs rather to a larger work than this. The student is referred to Fresenius, Wanklyn, Frankland, and others. A few remarks only will be made to show what any ordinary student might say with regard to a water under examination—only in a qualitative point of view. The operations for the quantitative analysis of such waters are not such as can readily be executed, and (if the writer may be allowed to express an opinion) the results are utterly unsatisfactory, as not showing in the slightest degree the cause of the wonderful medicinal action of these waters. Hence it has not been thought desirable to introduce into such a small work any elaborate method of analysis.

Most waters in Canada contain  $\text{CaCO}^3$ , simply dissolved in  $\text{H}^2\text{O}$ ; Lakes Superior, Michigan, Erie partly; Ontario also partly. The lower lakes contain the carbonate dissolved partly in  $\text{CO}^2$ . Most spring waters are similar; you will find scarcely one that does not give a precipitate on boiling for fifteen minutes.

Our Canadian waters are generally solutions of chlorides of calcium, magnesium, sodium—rarely with sulphate of magnesium; but sulphate of calcium occurs in greater or smaller quantity in almost all. Iodides and bromides occur in some; in a very few the iodine is detectable at once by  $\text{PdCl}^2$  test or others, generally in such small quantity that a gallon or more of the water has to be experimented on.

## ON RENDERING SUBSTANCES INSOLUBLE IN WATER SOLUBLE.

1. Substances having a metallic lustre ; metals and many ores.
2. Substances having no metallic lustre ; salts, &c.

### I.

#### SUBSTANCES HAVING A METALLIC LUSTRE.

Many metals, such as Fe, Zn, Cd, Al, Sn, are more or less easily soluble in warmed dilute HCl. If dilute acid has no action, stronger may be employed, and some sulphides are dissolved, such as those of Zn, Pb, Bi, Sb, with evolution of  $H^2S$ , detected by smell or action on lead paper. Of course, the substance should be in fine powder, and the solution heated until all  $H^2S$  is evolved, before dilution. If HCl has no action, a fresh portion should be treated with dilute or strong  $HNO^3$ ; almost all metals and sulphides, excepting Au, Pt, and  $HgS$ , will be acted on with evolution of red fumes ; the presence of S may be detected by the white precipitate formed in the solution by  $BaCl^2$ , owing to  $H^2SO^4$  produced. Some metals do not dissolve, but give a white insoluble oxide, such as Sn and Sb, after dilution with  $H^2O$ . PbS will also give, after long boiling, a white residue of  $PbSO^4$ , soluble in hot HCl, or in ammonium acetate, in which solution the Pb may be easily detected. If the metal which gave a white residue is brittle—Sb ; if malleable and rather difficultly soluble in HCl—Sn. The solution, if obtained, may be examined in the usual manner. If insoluble in either of the above named acids, a mixture of the two must be tried, when Au and Pt will be dissolved, and also  $HgS$ . Appropriate tests can then be applied.

### II.

#### BODIES NOT HAVING METALLIC LUSTRE.

The examination of silicates does not come within the scope of this small pamphlet ; but some are decomposed when in fine powder by HCl, and a jelly formed. On evaporating to dryness, the  $SiO^2$  becomes insoluble, and on treating with a few drops of HCl and  $H^2O$ , a solution of the bases will be obtained. The residue of  $SiO^2$  may contain Titanic acid. (See Blowpipe Tests.) Many are not so acted on, and must be fused for half an hour with  $Na^2CO^3$  or  $KN^2CO^3$ , the mass dissolved in  $H^2O$  and HCl, evaporated to dryness, the residue moistened with HCl, after a time  $H^2O$  added,

filtered, the residue is the  $\text{SiO}^2$  (?  $\text{TiO}^2$ ), and the filtrate contains all the bases. This applies principally to the ordinary earthy silicates. Other methods, especially for determining the alkalis by  $\text{HF}$ , or by heating with calcium carbonate and chloride, cannot be described here.

A great many salts of the heavier metals, which are insoluble, or nearly so, in boiling water, are readily acted on by  $\text{Am}^2\text{S}$ , forming metallic sulphide, and ammonium salt of the acid. Some may of course be dissolved entirely, but those cases are exceptional. The salts of zinc, aluminum, chromium and the alkaline earths will not be perceptibly altered. The residue after washing can be dissolved in appropriate acid, and the metal detected. The  $\text{Am}$  solution may be evaporated, mixed with water, filtered, and the acid discovered.

Some salts are not very easily acted on by  $\text{Am}^2\text{S}$ , such as the chromates; but these are all soluble in acids, recognised moreover by their colour, and by giving a green solution when boiled with  $\text{HCl}$  and alcohol or  $\text{SO}^2$ . The solution will contain  $\text{Cr}^2\text{O}^3$ , and the residue may contain  $\text{PbCl}^2$ ,  $\text{Hg}^2\text{Cl}^2$ , which are easily recognised as already described. The earthy oxides could of course be easily detected in such a solution.

The  $\text{PbSO}^4$ , sometimes met with as an adulterant, is soluble in hot  $\text{HCl}$ , and also in ammonium acetate, in which the metal can be detected.

Most of these salts can be decomposed by boiling with  $\text{Na}^2\text{CO}^3$ , when an oxide or carbonate remains, and the solution contains the acid; more especially by fusion, but  $\text{Hg}$  might and would be driven off at a high temperature. As instances may be mentioned, among the more common salts,  $\text{PbI}^2$ ,  $\text{PbCl}^2$ ,  $\text{Hg}^2\text{Cl}^2$ ,  $\text{HgI}^2$ .

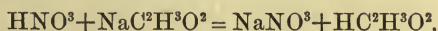
$\text{PbI}^2$  and  $\text{PbCl}^2$  as well as  $\text{PbBr}^2$  can be dissolved out by much boiling water, and the metal and radical detected in the solution; for the latter two boiling or fusing with  $\text{Na}^2\text{CO}^3$  for detection of radical would be required. The metal easily detected in the chloride of  $\text{Hg}$  by dry test, but in the iodide by solution in aqua regia, precipitation by  $\text{H}^2\text{S}$ , &c., &c.

The sulphates of barium, strontium and calcium are more or less insoluble in water or acids. They may be examined by boiling the fine powder with strong solution of  $\text{Na}^2\text{CO}^3$  for a considerable time, filtering, washing out, and testing the filtrate, after acidifying with



$\text{HNO}^3$ , with  $\text{BaCl}^2$  for  $\text{H}^2\text{SO}^4$ . The residue on filter, if drenched with  $\text{HCl}$ , will give a solution in which the base may be detected. The same may be effected by fusion with alkalic carbonate in a crucible. But for mere qualitative experiments, Bloxam's deflagrating mixture is much to be recommended: this is one part finely powdered charcoal, with six parts pure  $\text{KNO}^3$ ; the supposed sulphate, mixed with two parts  $\text{Na}^2\text{CO}^3$ , is mingled with three or four parts of the deflagrator, and burnt on a porcelain or clean iron plate. The whole mass takes fire, fuses, and causes a very complete decomposition. The mass can be pounded, boiled with water, and the tests applied as above.

Apatite is a rather abundant mineral in some parts of Canada. The above process might be adopted, but is not necessary. The following simpler plan may be used. The finely powdered mineral is dissolved in water with as few drops of  $\text{HNO}^3$  as possible; in testing for lime and phosphoric acid, very little  $\text{HNO}^3$  should be added; in examining for rock matter, a larger quantity of that acid, or  $\text{HCl}$ . A portion of the nitric solution, filtered if necessary, is mixed with excess of ammonium molybdate and warmed; a yellow precipitate indicates phosphoric acid. Another portion is mixed with  $\text{AgNO}^3$ , and *carefully* neutralised by  $\text{NH}^3$ ; a yellow precipitate of  $\text{Ag}^3\text{PO}^4$ , mixed with tribasic calcium phosphate, indicates the acid. Another portion may be carefully neutralised with  $\text{NH}^3$ , the precipitate of  $\text{Ca}^3 2\text{PO}^4$  dissolved in  $\overline{\text{Ac}}$ ; to half of this  $\text{AgNO}^3$ , when added, produces the above yellow precipitate; in the other, the addition of ammonium oxalate, or a little oxalic acid, produces a white precipitate. This test cannot be applied directly to the nitric solution, as calcium oxalate is soluble in  $\text{HNO}^3$ , but not in  $\overline{\text{Ac}}$ ; so that even in the nitric solution the addition of a considerable quantity of sodium acetate will cause the precipitate, because the nitric acid unites with the sodium, and acetic acid is set free.



This is a very useful plan for detecting calcium in many cases. Calcium oxalate, which not unfrequently occurs as urinary calculus or deposit, may be detected by dissolving in  $\text{HCl}$  or  $\text{HNO}^3$ , and reprecipitating by addition of sodium acetate. See further under Calculi.



## POISONS.

Without entering into any discussion as to the meaning of the word poison, it may be said that all substances, when consumed in improper quantities, act as such. In the following pages only those will be considered which, when administered or applied in small or moderate quantities, produce injurious effects, either mechanically or physiologically. In detecting these, more especially arsenic, the difficulty is not so much in the process, as in proving that the materials employed do not themselves contain the substance sought for. A few of the reagents required in such investigations are mentioned in the following pages, with modes of purification.

Some other substances are introduced which can scarcely be ranked among poisons, but which may produce injurious effects, more especially if impure, and which may conveniently be treated of under this head.

In many cases, where the symptoms accompanying the illness or death have been described, the examinations of the chemist are rendered comparatively easy, if it is necessary only to seek for the cause thereof. Thus, if a person lives for hours after a dose has been administered, it is not probable H<sub>2</sub>Cy was used; if he dies in a minute or so, probably H<sub>2</sub>Cy; if tetanic convulsion, strychnine; if narcotism, morphine; and so on.

But if no symptoms are detailed, the examination becomes more difficult. The following plan may be suggested as applied to the body submitted, say, the contents of the stomach. These being introduced into a beaker, may be treated as described under Arsenic, the liquid poured into a retort, and the washings also. The distillation may now be effected as recommended by the writer; the first products may contain ether, alcohol, nitrobenzol, or prussic acid, the latter passing off very early in the operation. Hence the first portions should be collected separately. At a later period, other bodies, such as carbolic acid, phosphorus, &c., may pass over. (See Phosphorus). If the liquid has been mixed with a little tartaric acid, no volatile alkaloids could distil over. The liquid in receiver examined for above poisons; the liquid in retort filtered, and filtrate examined for oxalic acid, or a portion of it; the remainder for alkaloids, as described. If none are found, all the residues may be evaporated to a syrup, and treated as described under Arsenic.

It is scarcely necessary to add that the contents of the stomach must be examined for mechanical irritants, such as pounded glass, roots, or leaves of plants, more or less poisonous, crystals of arsenious acid, corrosive sublimate, cantharides elytra, &c., &c.

#### SULPHURIC ACID.

This acts by its corrosive (dehydrating) action. It is not now so much used as in former times; if thrown on to a person, parts of the clothing on which the acid has fallen may be boiled with water, the solution filtered and tested for  $\text{H}^2\text{SO}^4$  in the usual manner. The contents of the stomach must be examined in same way, bearing in mind that slight traces of sulphates will always be detected. If chalk or magnesia has been administered as antidote, the liquid may not be acid; and if chalk has been employed, the insoluble sulphate must be examined as described under Insoluble Substances.

#### NITRIC AND HYDROCHLORIC ACIDS

Are seldom used as poisons, but accidents may happen from incautious use of them. The ordinary tests can be applied, bearing in mind that chlorides, and perhaps free  $\text{HCl}$ , are always present in the gastric juice; it is said, 1.7 in 1000 of gastric juice.

#### ARSENIC.

The chemist may be called upon to detect this substance in solution, such as Donovan's or Fowler's; in a solid, such as arsenious or arsenic acid, arsenites or arsenates, fly powder (arsenic), sulphides and arsenides. It may also in one of its forms be mixed with organic matter, as in food, the contents of the stomach, vomited matter, fæces, urine, the liver, and other parts of the body. It may also be necessary to look for it in wall papers, many of which, beside the green, it has lately been stated, contain arsenious acid, which has been added for brightening the colour; moreover, it may exist in many textile fabrics, to which it has been applied directly as a colouring matter, or indirectly as existing in certain aniline colours. Green wreaths frequently contain arsenic.

If a solution has to be examined, the usual tests can be applied; if in Donovan's solution, the arsenic and mercury precipitated by  $\text{H}^2\text{S}$ , the precipitate washed, treated with  $\text{NH}^3$  or  $\text{Am}^2\text{S}$ , the solution evaporated, and the residue treated as described hereafter.

If a solid has to be examined it may be heated in a small tube, when a brown or white crystalline sublimate will be formed, the octahedral crystals of the latter being easily recognisable under the microscope. Other tests as described hereafter.

The solid may be an arsenite or arsenate insoluble in water, or it may be arsenious acid mixed with ferric oxide, or other substance of a mineral nature. In that case it should be heated in a reduction tube with sodium carbonate and potassium cyanide or charcoal, when a ring will be formed of a brown colour or with a metallic lustre. Sulphides of arsenic may be treated in the same way, but it is better to boil repeatedly with fuming  $\text{HNO}_3$ , until all the sulphur is oxidised, evaporate to dryness, and heat the residue with the above mixture. Insoluble arsenites or arsenates may also be dissolved in dilute  $\text{HCl}$  or  $\text{HNO}_3$ , precipitated by  $\text{H}_2\text{S}$ , the arsenical sulphide dissolved in  $\text{NH}_3$ , evaporated and the process conducted as above; this however is seldom necessary. The sulphide obtained in any of these cases may easily be converted into arsenite of potassa by dissolving in caustic potassa, boiling with bismuth hydrate, and filtering from the bismuth sulphide produced,  $\text{As}_2\text{S}_3 + \text{BiO}_3 = \text{As}_2\text{O}_3 + \text{BiS}_3$ .

If mixed with organic matter, as in urine,  $\text{HCl}$  should be added, and the As precipitated as sulphide by  $\text{H}_2\text{S}$ .

If suspected in the contents of a stomach, the whole should be rinsed out with water into a beaker, allowed to stand for a few minutes, the liquid poured off, and the operation repeated till most organic matter is removed. Any white, sandy and crystalline or dark coloured substance remaining may be examined by the liquid or dry tests; in the latter case, the substance must be previously dried.

Whether any such residue has been found or not, the whole liquid should be evaporated over a water bath until reduced to a thick mass, and treated as hereafter described, either by itself or with the stomach cut up into small pieces. The liver, spleen and intestines, or a portion of them, should be cut up and examined in the same manner. The examination of the liver and spleen, &c., is of much importance, as it sometimes happens that all the poison has been eliminated from the stomach, and the presence of it in those organs is a strong proof that it was taken into the system during life. It has been objected that if a body has been buried in a soil containing arsenic, some may have been absorbed, but this is not of much value, and may to a



certain extent be got over by examining the internal portion of the liver, and of course by testing the soil.

The different portions are now placed in clean porcelain dishes, covered with HCl of specific gravity of about 1.11 (obtained by mixing together equal bulks of the strong acid and water), the freedom of which from arsenic must have been previously ascertained. The operations for dissolving and destroying the organic matter are conducted as follows :

I. The mixture is heated for some time (15 minutes) over a water bath or common saucepan and powdered potassium chlorate added in portions of about fifty grains from time to time, allowing the effervescence to subside after each addition ; of course the whole should be well stirred, and the operation continued until the whole of the solid matter is disintegrated and converted into a yellow flocculent substance. The liver is always harder to dissolve than the other parts and it may be necessary to add more HCl, if the addition of the chlorate causes no effervescence. The operation must of course be conducted in the open air or under a shed with a good draught. When all has been thus converted, an equal bulk of water is added and the heating continued till all the chlorine is expelled.

It has been recommended to conduct the above operation in a retort, to collect in an attached receiver any  $\text{AsCl}_3$  that might pass over ; this renders the process more troublesome and is not necessary if the temperature be kept below  $100^\circ \text{C}$ .

It has been stated that in cases where all the softer parts of a body have been decomposed, arsenic has been found in the pelvis and adjoining vertebræ. The operation would be the same, HCl being added till all is dissolved.

II. The whole is now filtered through paper or linen, free from arsenic ; and if too bulky, may be evaporated down to about a pint. It does not appear, however, that this operation is desirable, being tedious, and likely to entail a slight loss of arsenic.

III. Through the hot liquid a current of washed  $\text{H}_2\text{S}$  is now passed for several hours, the precipitate formed thrown on to a filter and well washed. A little more of the precipitate may form on standing ; if distinctly yellow, it is probably arsenic alone ; but if orange or dark coloured, the precipitate may contain arsenic masked by antimony or some other metal. Frequently a faint light coloured



precipitate is formed, when no arsenic is present, from the action of some residual chlorine on the  $\text{H}^2\text{S}$ .  $\text{Cl}^2 + \text{H}^2\text{S} = \text{Cl}^2\text{H}^2 + \text{S}$ .

During the oxidation, the arsenic or arsenious acid will have been converted into arsenic acid; and it has been recommended to reduce this to arsenious by saturating with sulphur dioxide and heating to expel excess,  $\text{As}^2\text{O}^5 + 2\text{SO}^2 = \text{As}^2\text{O}^3 + 2\text{SO}^3$ . The object is to render the precipitation more easy; this complicates the process, and can be avoided by continuing the current of  $\text{H}^2\text{S}$  for a long time. The first action of the  $\text{H}^2\text{S}$  seems to be the same reduction as effected by  $\text{SO}^2$ .

IV. The precipitate on filter, after being well washed, is drenched with  $\text{NH}^3$ , if adhering it may be rubbed down with a feather; the filtrate is received in a porcelain capsule, evaporated on water bath to dryness and treated with successive portions of fuming  $\text{HNO}^3$  until all the sulphur is dissolved. This however is not absolutely necessary; by the operation most of the organic matter will have been destroyed but a small portion may remain. In order to remove it the following process is adopted.

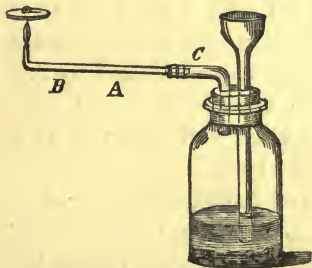
V. The sulphide having been boiled with  $\text{HNO}^3$  will contain, if As be present,  $\text{As}^2\text{O}^5 + \text{H}^2\text{SO}^4$ , with perhaps trace of organic matter. The solution is now neutralised by pure  $\text{Na}^2\text{CO}^3$  or  $\text{NaHCO}^3$ , evaporated to dryness and the residue thrown, in small portions at a time, into a crucible in which one or two drachms of pure sodium nitrate are kept in fusion. The operation should be so conducted that the mass in the crucible remains perfectly fused and clear after each addition. By this all organic matter is destroyed or converted into carbon dioxide. Hence we have in the fused mass, if As be present, the following substances:  $\text{Na}^3\text{AsO}^4$ ,  $\text{Na}^2\text{SO}^4$ ,  $\text{NaNO}^3$ ,  $\text{Na}^2\text{CO}^3$ , if the latter be used in excess.

If the fused mass be boiled with  $\text{H}^2\text{O}$ , the arsenate, &c., will be dissolved, but antimony (if present) remain undissolved as antimonate, hence that metal is thus eliminated; if treated with  $\text{H}^2\text{SO}^4$ , as described in VI., of course Sb would be dissolved.

VI. As Marsh's test will not succeed in presence of  $\text{HNO}^3$ , it is now necessary to get rid of that substance. That is done by heating the fused and cooled mass in a rather large crucible with pure  $\text{H}^2\text{SO}^4$ , until quite dissolved, and no red fumes, but only white of  $\text{H}^2\text{SO}^4$  are evolved. The residual mass will be hydric sodium sulphate,  $\text{NaHSO}^4$ , mixed with sodium arsenate.

VII. The residual mass is now dissolved in water, introduced into a measured burette, diluted with water to make it up to a certain quantity, say 100 CC, well mixed and a small portion employed for the next experiment.

VIII. A two-necked bottle is provided with a thistle-funnel in one aperture reaching to near the bottom, and a gas delivery tube in the other; this tube should be bent at right angles a little above the bottle and for careful experiments should be connected with a drying tube filled with  $\text{CaCl}_2$ . The arrangement, which is very simple, may best be explained by a diagram representing the simplest arrangement. By attaching a drying tube with  $\text{CaCl}_2$  or  $\text{H}^2\text{SO}^4$  to *C*, and adding thereto the bent tube marked *B A*, the apparatus may be made more complete. Probably pumice stone soaked in strong  $\text{H}^2\text{SO}^4$  is a better drying material than any other.



Attached to the  $\text{CaCl}_2$  by means of a flexible joint, is another tube about ten or twelve inches long and quarter of an inch wide, drawn out to a coarse point and bent at right angles. This can be supported on a stand, strengthened perhaps by some copper gauze which will prevent its bending when heated. Acid is now poured on to the zinc and gas evolved till all the air is expelled from the apparatus. Heat must not be applied at *A* until this is the case, to avoid an explosion. The best way of ascertaining this is to hold a small test tube for a few minutes over the terminal point and after removal applying a light to the mouth, held of course downwards. If a slight explosion occurs, more gas must be evolved until the hydrogen burns quietly in the tube, and the issuing gas can be inflamed by it. Heat is now applied at *A*, and continued for ten or fifteen minutes; if no dark or metallic ring is formed at *B*, the materials may be considered pure.

IX. The suspected liquid is now introduced through the funnel, and if arsenic be present a ring will speedily be formed. If the quantity of arsenic be small, the whole of the  $\text{AsH}^3$  will be decomposed, but if large, or the current be too strong, some will pass off unchanged and may be burnt at the point. Spots can be obtained on porcelain plates or crucible covers by holding them in the blueish white flame. The point may also be bent down and the gas passed into an ammoniacal solution of  $\text{AgNO}^3$ . Metallic silver will be deposited and ammonium arsenite remain in solution.

For coarse or ordinary experiments the above apparatus may be dispensed with and the experiment made with an 8-oz. phial with a short straight tube, with a not too fine point and ground obliquely below. The arsenic may be introduced, after testing the purity by a plate by blowing out the flame, removing the cork, &c. The plates should not be held too long in the flame as the arsenic might be volatilised. If antimony has not been eliminated in preceding operations, the ring or spots may be caused by that metal. The spots will be, if at all dense, black and velvety, and the ring form very close to the heated part *A*, while spots of As are brown or metallic and farther from heated part.

X. Further means of distinguishing are as follows :

The rings volatilise easily when heated .....	As
They volatilise only partially, and when strongly heated, yield globules of metallic antimony.....	Sb
Heated in current of $\text{H}^2\text{S}$ , become yellow .....	As
Heated in current of $\text{H}^2\text{S}$ , become orange .....	Sb
The yellow ring is not acted on by $\text{HCl}$ .....	As
The orange ring is dissolved by $\text{HCl}$ .....	Sb
The ring or spot drenched with solution of $\text{KIO}^3$ is dissolved with faint reddish cloud.....	As
Is not acted on at all by $\text{KIO}^3$ .....	Sb
The ring or spot dissolves in hot $\text{HNO}^3$ , and the solution, if evaporated to perfect dryness, gives a red precipitate or colour with $\text{AgNO}^3$ ....	As
Gives no colour .....	Sb

Spots of As produced on platinum foil disappear on heating, with garlic colour; spots of Sb do not. By heating the crucible cover on which a spot has been formed with a microscope slide over it, octahedral crystals of  $\text{As}^2\text{O}^3$  are produced. Sb gives no crystal under such circumstances.

There are many other tests, but the above are sufficient.



XI. Arsenic having been detected, it may be requisite to determine the quantity. The remainder of solution is treated with  $\text{SO}^2$ , to reduce  $\text{As}^2\text{O}^5$  to  $\text{As}^2\text{O}^3$ , boiled to expel  $\text{SO}^2$ , precipitated by  $\text{H}^2\text{S}$ , the  $\text{As}^2\text{S}^3$  collected on a tared filter, washed, dried and weighed; one hundred parts represent 80.4 of  $\text{As}^2\text{O}^3$ . Or the solution from VII. may be mixed with  $\text{MgSO}^4$  and  $\text{NH}^3$ , allowed to stand twenty-four hours, the precipitate treated as above, but washed with dilute  $\text{NH}^3$ ; one hundred parts of the  $\text{AmMgAsO}^4$  represent 52.1 of  $\text{As}^2\text{O}^3$ .

XII. Fleitman recommends boiling the liquid or solid containing arsenic with zinc or magnesium and potassium hydrate.  $\text{AsH}^3$  is evolved, which can be detected by giving a dark stain on paper soaked in  $\text{AgNO}^3$ . No change is produced by  $\text{SbH}^3$ .

Davy recommends the use of sodium amalgam, applied to the liquid or moistened solid without heating, and testing as above. This process is very simple and wonderfully delicate. Davy detected  $\frac{1}{1000000}$ th of a grain in a short time, and  $\frac{1}{1000000}$ th in fifteen minutes; other observers have detected still smaller quantities.

Some chemists simply digest the animal or organic substance with  $\text{HCl}$ , filter, and apply Reinsch's test, *i.e.*, metallic copper. There is not much certainty of thus extracting all the arsenic.

Where large quantities of organic matter have to be operated on, it has been recommended to char the mass with  $\text{H}^2\text{SO}^4$ , or to digest with  $\text{HNO}^3$ , convert into  $\text{NaNO}^3$ , and deflagrate so as to form arsenate. In both these cases there is a danger of loss.

Other methods than the one fully described above, invented originally by Duflos and improved by Otto and others, have been proposed, but may be omitted from this small work. It has been remarked, the difficulty of the process does not lie in the detection of the arsenic, so much as in the necessity of proving its absence from the reagents employed.

NOTE.—In using the  $\text{NaHg}$  test, the solution must be made alkaline previously, as  $\text{Sb}$  will produce similar results in acid solutions; but the stains may be distinguished by soaking in  $\text{Am}^2\text{S}$ , yellow or orange stains produced, also by action of  $\text{HCl}$  thereon.

## PURE SULPHURIC ACID.

### FOR TESTING FOR ARSENIC.

In toxicological investigations this acid must be perfectly pure; it seldom contains  $\text{HCl}$  or  $\text{HNO}^3$ ; sometimes  $\text{SO}^2$ ; but frequently  $\text{As}^2\text{O}^3$ . From all these it may be freed by a process which is pro-



bably preferable to all others, as it gives a concentrated acid. Place into a tubulated retort of sufficient capacity a quantity of commercial or so called pure  $\text{H}^2\text{SO}^4$ , with receiver attached. Arrange a flask containing mixture for evolving  $\text{HCl}$ , four parts by weight  $\text{NaCl}$ , seven parts  $\text{H}^2\text{SO}^4$ , and two parts  $\text{H}^2\text{O}$ . Fasten into the flask a cork with a tube bent twice at right angles, or the longer arm at somewhat less, fitted into a cork which is adapted to the tubulure of the retort, so that the longer arm passes down to the bottom of the  $\text{H}^2\text{SO}^4$ . Heat this by spirit lamp, and drive the  $\text{HCl}$  through it, until the distillate which collects in receiver shows no trace of yellow precipitate with  $\text{H}^2\text{S}$ . The reaction is this:  $\text{As}^3\text{O}^3 + \text{H}^6\text{Cl}^6 = \text{As}^3\text{Cl}^3 + \text{H}^6\text{O}^3$ ; the former being volatile passes over, and thus all arsenic is removed. Detach the  $\text{HCl}$  flask, and heat the  $\text{H}^2\text{SO}^4$  until a portion of it, diluted with water, gives no precipitate with  $\text{AgNO}^3$ . In this way, half a pound of  $\text{H}^2\text{SO}^4$  may be freed from  $\text{As}^3\text{O}^3$ ,  $\text{HCl}$  and  $\text{HNO}^3$  in an hour at most.

#### HYDROCHLORIC ACID.

★ The pure acid of commerce is generally free from  $\text{As}^3\text{O}^3$ ; the common is not. It can be purified by diluting with equal bulk of water, passing  $\text{H}^2\text{S}$  through for some hours, allowing to stand in warm place till all  $\text{H}^2\text{S}$  is driven off, and filtering. This is sufficiently strong for the purpose above mentioned; this is perhaps easier than making the gas from mixture mentioned in above paragraph, passing into a bottle containing a small quantity of water, and thence into more water, kept cool, as long as any gas is evolved.

#### NITRIC ACID

Is generally pure, but may contain some  $\text{HCl}$ ; if heated, this will be decomposed, and the residue may be considered as sufficiently pure, when, if diluted with water, it gives no precipitate with  $\text{AgNO}^3$ .

#### SODIUM BICARBONATE

★ Is often very pure. Chlorides should be eliminated. This may be done by filling a filter with packed salt, letting pure water run through until the filtrate, neutralised with  $\text{HNO}^3$ , gives no opalescence with  $\text{AgNO}^3$ . The residue when dried is pure bicarbonate.

#### SODIUM NITRATE.

By neutralising pure  $\text{HNO}^3$  with the above, and evaporating to dryness.

## POTASSIUM CHLORATE

Is almost always pure.

## ZINC.

Commercial metal is often free from arsenic, but had better be purchased from persons who manufacture it in a pure state. Use of sodium amalgam obviates this difficulty to a certain extent.

## ANTIMONY.

**X** If this metal be present in the liquid mentioned in par. I. under Arsenic, its presence, if unaccompanied by dark coloured sulphides, will be immediately recognised by the orange red precipitate formed by  $\text{H}_2\text{S}$ , unless present in very small quantity. The precipitate may contain arsenic sulphide, which can be removed by  $\text{NH}_3$ . If dark coloured sulphides be present, that of Sb can be extracted by  $\text{Am}^2\text{S}$ , and after evaporation treated with  $\text{NH}_3$  to remove As. Or it may be fused with  $\text{NaNO}_3$ , and the arsenate extracted by water. The residual antimonate, or the sulphide, may be dissolved in  $\text{HCl}$ , and the usual tests for Sb applied.

## TIN.

Cases of poisoning by salts of this metal are usually accidental, some of them being employed in dyeing. The precipitate with  $\text{H}_2\text{S}$ , in liquid obtained as in par. III., will be of a dirty yellow colour. Fused, when dry, with  $\text{Na}_2\text{CO}_3$  and  $\text{KCy}$ , a bead of tin will be obtained, which will dissolve in  $\text{HCl}$ , and the solution will give the reaction of stannous salts.

## BISMUTH.

Although the salts of this metal are seldom used as poisons, yet as some are used as cosmetics, being comparatively harmless, it may be necessary to distinguish them from lead carbonate, which is sometimes most improperly used for that purpose. The salt, whatever it may be, can be dissolved in  $\text{HNO}_3$ , and the usual tests applied.

## ZINC.

Salts of this metal occasionally produce fatal results, generally through accident. The liquid filtered off after treatment with  $\text{H}_2\text{S}$ , par. III., must be neutralised with  $\text{NH}_3$ , and perhaps a little  $\text{Am}^2\text{S}$  added. If zinc be present, a light coloured greenish (iron) precipitate

will be formed ; this when well washed can be dissolved in  $\text{HNO}^3$  warmed to peroxidise any iron, precipitated by excess of  $\text{NH}^3$ , filtered, and the filtrate tested for zinc.

#### MERCURY.

The precipitated sulphide is black, and after being well washed is insoluble in  $\text{HNO}^3$ , but soluble in a mixture of that acid with  $\text{HCl}$ . The solution can be tested for mercuric salts ; or the dried precipitate may be heated in a tube with  $\text{Na}^2\text{CO}^3$ . Very small traces of this metal may be detected by placing in the dilute solution a piece of gold wire, round which some strips of tin foil have been wound. A gray coating will be formed, and if the gold wire be dried and heated in a tube, a sublimate of mercury will be obtained. It must be remembered that mercurous chloride (calomel) would be converted by the above process into mercuric chloride. On one occasion the writer found small crystalline particles of corrosive sublimate adhering to the coats of the stomach ; these, heated with  $\text{Na}^2\text{CO}^3$ , gave at once the characteristic reaction.

#### COPPER.

The precipitated sulphide is black, insoluble in  $\text{NH}^3$ , soluble in  $\text{HNO}^3$ . To this solution the ordinary tests can be applied.

Copper arsenite, when boiled with  $\text{HKO}$ , gives a red insoluble powder of cuprous oxide, and a solution which contains arsenic acid. All compounds, when moistened with  $\text{HCl}$  and exposed to the blow-pipe flame, produce a blue or green colour.

#### LEAD.

The precipitated sulphide is black, insoluble in  $\text{NH}^3$ , soluble in hot, moderately strong  $\text{HNO}^3$ , leaving some  $\text{PbSO}^4$  as a white residue. In this, or in the solution, the metal can be detected by the usual tests.

#### PHOSPHORUS.

Accidental poisoning from this substance sometimes occurs, arising from use of it in rat-poison and in matches.

The substance suspected to contain phosphorus should be boiled in a flask or retort, perhaps with the arrangement mentioned under  $\text{HCy}$ , and the vapours conducted through a bent tube into a cooling apparatus, either perpendicular, *i.e.*, a cylinder with hole in the base, or a glass Liebig's condenser, that can be easily constructed.



This being cooled in the ordinary way, a light will be seen where the vapours begin to be condensed. Of course the experiment must be made in a dark room. If a receiver be attached, small particles of P will collect in the water contained therein. These may be examined directly for P, or boiled with  $\text{HNO}_3$ , and examined by appropriate tests for  $\text{H}_3\text{PO}_4$ . The liquid portion may also be evaporated with  $\text{HNO}_3$ , and examined in the same manner.

The very beautiful test depending on the green colour communicated to the flame of hydrogen by presence of phosphorus need not be mentioned here. (See Otto, *Ausmittlung der Gifte*, &c.)

A very simple and easily executed test is to boil the suspected substance with water in a flask, and fasten on to the cork, lightly fitted in or provided with an exit, two papers; one soaked or moistened with  $\text{AgNO}_3$ , the other with  $\text{PbAc}$ . If P be present, on boiling, the Ag paper will be blackened from separation of metal or  $\text{AgP}$ , the lead paper not. If  $\text{H}_2\text{S}$  be present, both will be blackened. This test will show the phosphorus in the end of a single common match with the greatest ease. In the case of contents of a stomach, undergoing partial decay, the distillation (Mitscherlich) process had better be adopted, and an infinitesimal portion of the condensed solid in the distillate may be used for above experiment.

## ORGANIC POISONS.

This group contains many substances that act as virulent poisons, but others that can scarcely be ranked as such are introduced here for convenience.

### OXALIC ACID.

Accidents sometimes occur from the use of this substance instead of magnesium sulphate. The dry acid itself is easily distinguished from Epsom salts, by the striated and partly opaque appearance of the crystals, by its acid reaction, and by its perfect volatility when heated. Acid oxalates are sometimes sold under the name of "salt of lemons" (citric acid), easily distinguished by blackening very slightly when heated, leaving a carbonate, and giving a precipitate with salts of calcium even in the cold. When mixed with organic matter, as in the contents, a clear solution should be obtained, precipitated by lead acetate, the precipitate washed, decomposed by  $\text{H}_2\text{S}$ , filtered, and the filtrate examined for oxalic acid.



## HYDROCYANIC ACID.

The acid and the cyanides are all poisonous; hence accidents from use of KCy by photographers, and of oil of bitter almonds, which when crude contains HCy. Laurel and bay leaves, peach kernels, and even apple pips have been found to yield HCy when distilled, especially after having been allowed to stand with lukewarm water for some time. Nitrobenzol, essence of mirbane, is poisonous, has smell of oil of bitter almonds, but contains no HCy.

The special tests for HCy have already been described. On opening the brain or body of a person or animal recently killed by HCy, the odour is quite discernible, but rapidly disappears owing to the volatility of the acid. It is recommended to distil the contents of the stomach with some acid if a cyanide is suspected; the writer advises to heat the same in a retort gently, and to pass steam from a flask into it; in this way every trace of HCy can be driven off without any danger of charring or bumping, and appropriate tests applied to the condensed distillate. In this way HCy was detected after thirteen days' interment.

The other direct tests with  $\text{AgNO}_3$  and  $\text{Am}^2\text{S}$ , as described under HCy, may be applied to the distillate or contents.

## DETERMINATION OF STRENGTH OF HYDROCYANIC ACID.

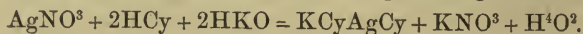
As this preparation is very apt to deteriorate, it is often of importance to determine its strength. Two methods may be adopted.

A known weight, say 100 grs., of the acid is *fully* precipitated by  $\text{AgNO}_3$ , the resulting AgCy collected on a tared filter, washed, dried and weighed. The amount of AgCy, divided by 5, gives quantity of HCy very nearly.

$$\text{HCy} = 1 + 26 = 27; \text{AgCy} = 108 + 26 = 134.$$

Another more rapid plan is to weigh out 54 grains of the acid, add excess of HKO, and if the latter be pure, a little NaCl. Then add gradually to this a solution of 170 grs.  $\text{AgNO}_3$  in 1000 grs.  $\text{H}_2\text{O}$ , until after stirring a permanent precipitate is formed. Each division employed will indicate 0.1 per cent. of HCy.

When KCy acts upon any salt of Ag, the soluble double cyanide KCyAgCy will be formed, and no chloride can be produced until the whole of the Cy has been employed in producing this compound.



$$\text{AgNO}_3 = 108 + 14 + 48 = 170; 2\text{HCy} = \text{H}^2 + \text{Cy}^2 = 54.$$

As burettes are divided into 100 parts, the whole amount if required would indicate 54 per cent. HCY, *i. e.*, 100 per cent.; but using a ten times weaker solution, that is, 17 grs.  $\text{AgNO}_3$  to the 1000, the whole would indicate 10 per cent., or 1 division 0.1.

Take 54 grs. of specimen, add HKO as above, and then the silver solution till permanent precipitate occurs. If 2.5 divisions have been used, 2.5 per cent. of HCY are indicated.

This solution of  $\text{AgNO}_3$  is useful in many cases of volumetric analysis.

#### AMYLIC ALCOHOL

1. In a liquid, such as a spirit, may be detected by diluting with an equal bulk of water, agitating with ether, pouring off the ethereal solution; on evaporation at low temperature the peculiar disagreeable smell may be recognised. This method is much more accurate than that of allowing the spirit to evaporate on the hand.

2. The amylic alcohol left by the ether may be warmed with potassium bichromate and sulphuric acid, when the peculiar odour of valerianic acid, &c., becomes noticeable.

#### ETHER

Should be perfectly neutral to test paper. Should have specific gravity of .720 nearly.

When ten measures are shaken up with ten measures of water, the ether should occupy not less than nine measures, ether being soluble in about ten parts of water.

#### ALCOHOL

Should be perfectly neutral to test paper. Ordinary strong alcohol should have a specific gravity of 0.83.

When it is required to ascertain the quantity of alcohol in any liquid, 4000 measured grains (or other quantity) should be distilled with careful cooling, until about one half is passed over; this is mixed with water until the original bulk is obtained. The specific gravity of this being taken at  $15^\circ\text{C}$  will show, on reference to table, p. 114, the percentage of absolute alcohol.

Before distilling, 15 or 20 grains of tannic acid should be added. To convert this into proof spirit, multiply by 2.04.

1. The substance suspected to contain alcohol must be distilled, if a thin liquid, by itself, with perhaps the addition of a little tannic

acid to prevent frothing, or if thick, by means of the apparatus described under Hydrocyanic Acid.

2. The distillate mixed with a few drops of potassium bichromate and HCl, or  $\text{H}^2\text{SO}^4$ , and heated, gives a green colour. Warmed with potassium hydrate, becomes yellow from formation of aldehyde. Methylated alcohol will become yellow at once on heating with HKO.

3. To a portion of the first distillate a few drops of solution of potassium hydrate are added, then solution of iodine in potassium iodide (not tincture of iodine), until a brown colour is produced, then more hydrate, until brown colour disappears. The solution, on standing or evaporation, deposits fine crystals of iodoform  $\text{CHI}^3$ , recognisable by odour and by form of crystals, either stellate, like triple phosphate, or hexagonal tables, like cystin.

Methylic and amylic alcohols do not give iodoform.

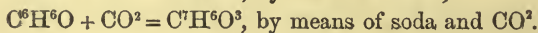
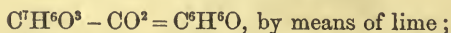
The product of the distillation should be neutral or only faintly acid. In distilling wines or beers, it is often strongly acid, owing to acetous fermentation having commenced. The acetic acid can of course be retained by using sodium carbonate instead of tannic acid.

For detection of aniline colours in wines, see under that head.

#### SALICYLIC ACID.

1. Solution gives fine purple colour with ferric chloride.
2. Dry acid, heated with soda lime or lime alone, evolves smell of phenol.
3. With sodium amalgam, the same.

Relation between phenol and salicylic acid :



#### NITROBENZOL

1. May be separated from its solutions by shaking with ether. This solution, poured off and allowed to evaporate, evolves the well known smell resembling oil of bitter almonds.

2. The ethereal solution may be reduced by zinc and hydrochloric acid, or by sodium amalgam. On addition of potassium hydrate and shaking with ether, aniline is extracted, and can be recognised by proper tests. See Aniline.

## CARBOLIC ACID—PHENOL.

1. Can be precipitated from its solutions, as in urine, by bromine water. The tribromo-phenol is filtered off and washed, gently warmed in a tube with sodium amalgam and water; the liquid poured off and acidulated, when the smell of phenol is given off. Some other bodies will however act in a similar manner.

2. Phenol in solution, boiled with mercurous nitrate, turns red; salicylic acid also.

3. With ammonia and chloride of lime, gives a blue or green colour.

4. Heated with nitric acid, gives carbazotic acid (trinitro-phenylic) which may be recognised by giving a yellow precipitate with potassium salts, or by giving dark purple (picramate) with ammonium sulphide.

Phenol may thus be distinguished and detected in creasote, which gives only oxalic acid.

5. By giving a green or blue colour with ferric chloride, while creasote gives brown.

A test for this substance (as well as alcohol) has lately been proposed by Davy, consisting in adding one drop of a very weak solution of the acid to four or five drops of a solution of about one part molybdic acid in ten parts strong and pure  $\text{H}^2\text{SO}^4$ . A yellow tint is produced, turning to brown, and finally purple (brownish purple). By addition of  $\text{H}^2\text{O}$  or exposure to air, the purple colour vanishes, but may be restored by addition of  $\text{H}^2\text{SO}^4$ . Very gentle heat assists the reaction. The test, when performed with the precautions advised in the Canadian Pharmaceutical Journal for August, 1878, seems to be very delicate. It is said that the carbohc acid in cows' urine can thus be detected.



## ALKALOIDS.

The general reagents for detection of almost all these bodies as a class, are as follows :

Ammonium molybdo-phosphate.

Sodium tungsto-phosphate.

Potassium cadmio-iodide.

Potassium mercuric-iodide.

These may be readily prepared, especially the last, in ordinarily well appointed laboratories, but the following are always available :

Potassium periodide, *i. e.*, solution of I in KI.

Dilute ferric chloride, with a few drops of solution of KI added. The writer's experience would lead him to prefer this, for its simplicity and sensitiveness, to all the others. The action of  $\text{Fe}^2\text{Cl}^6$  on KI is to set I free, which combines with the alkaloids forming Di or Ter iodides of a dark colour, more easily recognised than the white precipitates formed by the three first mentioned reagents.

## NICOTINE.



On evaporating ethereal solution, remains as a colourless or yellowish oil, having strong smell of tobacco.

Not evidently affected by chlorine.

Gives, with  $\text{PtCl}^4$ , and other reagents mentioned on previous page, the usual reactions.

It appears that the smoke from tobacco does not contain nicotine, but analogous compounds, such as parvoline,  $\text{C}^9\text{H}^{13}\text{N}$ , collidine,  $\text{C}^8\text{H}^{11}\text{N}$ , principally from cigars ; pyridine,  $\text{C}^5\text{H}^5\text{N}$ , principally from tobacco used in pipes. The latter seems to be more injurious.

## CONIINE.



The liquid alkaloid or amine, gives off when heated a very strong pungent and repulsive odour.

Chlorine water produces a white precipitate.

$\text{PtCl}^4$  produces no precipitate.

This alkaloid is contained in the roots of various species of conium, which are not unfrequently mistaken for parsnips, and the use of which has often led to serious accidents. The detection of the alka-

loid itself is usually attended with much difficulty. The presence of pieces of the root, and collateral circumstances, may often lead to a decision as to the cause of death, when resulting from coniine.

#### ANILINE.

$C^6H^7N$ .

Although this is not a natural alkaloid, it has been placed here as belonging to the class of volatile liquid amines, like the two preceding. Salts of aniline are well known to be injurious, and probably the poisonous effects of nitrobenzol are owing to the formation of this substance in the body.

Aniline gives purple colour with hypochlorites and various oxidising bodies.

With chlorine or bromine water, a dirty white precipitate.

With  $PtCl^4$ , no precipitate; hence differing from most bodies of this class.

With other above-mentioned reagents, gives no very characteristic reactions.

All the above volatile poisons may be separated from mixtures by the Stas process, or may be distilled over on boiling with strong alkali, separated from the distillate by shaking with ether, and evaporating the ethereal solution at low temperature. See Separation of Alkaloids.

#### FIXED ALKALOIDS.

Cannot be distilled over by means of water when by themselves; in presence of HKO, many of them give volatile alkaloids or amines such as morphine, codeine, cinchonine, &c.

#### MORPHINE.

$C^{17}H^{19}NO^3$ .

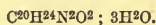
1. Precipitated from its solutions by HKO, easily soluble in excess.
2. Morphine and salts give a red colour with  $HNO^3$ , destroyed by reducing agents, such as  $SnCl^2$ ,  $SO^2$ , &c.
3. Dry salt or concentrated solution gives, with  $Fe^2Cl^6$ , a green colour; most characteristic test for morphine; succeeds best with freshly precipitated morphine.
4. Salts of morphine, added to solution of  $HIO^3$ , produce a yellow or brown colour, owing to separation of iodine, which may be detected by starch,  $CS^2$ , or other reagents.

Many other tests have been proposed for morphine, but the best is probably No. 3. The others have been tried, but found not available for general use.

It may be remarked that in attempting to extract the alkaloids according to Stas' process, it may be well to add the ether or chloroform before the addition of  $\text{NH}^3$ . Morphine, when once crystallised, is nearly insoluble in ether. This point has to be attended to when searching for morphine in organic mixtures.

In cases of poisoning by opium, the red colour produced by neutral  $\text{Fe}^2\text{Cl}^6$  (meconate of iron) is more certain than test for morphine, as meconic acid occurs only in opium. See methods of distinguishing this acid from acetic and sulphocyanic, after tables for acids.

### QUININE.



Alkalies and their carbonates produce in salts of this alkaloid a white precipitate, insoluble in excess of the reagent, becoming slightly crystalline after a time, easily soluble in ether. On shaking the mixture, a clear ethereal solution is obtained.

Hydric sodium-carbonate produces an immediate precipitate if the solution be rather strong (1 to 100), but only after a time if weak.

Chlorine or bromine water and  $\text{NH}^3$ , added to a solution of quinine, produce a fine green colour.

The same with a little  $\text{K}^4\text{Cfy}$ , added to solution of quinine, and then a few drops of  $\text{NH}^3$ , give a fine red colour, passing into brown, destroyed by acids, reproducible by  $\text{NH}^3$ . Concentrated  $\text{H}^2\text{SO}^4$ , heated with quinine or its salts, produces a yellowish colour, turning gradually to brown. Quinine itself, as well as its salts, is intensely bitter. Tincture of iodine, or the  $\text{KI}$  and  $\text{Fe}^2\text{Cl}^6$  solution, gives a dark coloured precipitate in the sulphate of the iodo-sulphate. This, if washed and dissolved in boiling alcohol, forms dark green tabular crystals, remarkable for their optical properties.

### CINCHONINE.



Alkalic hydrates produce a precipitate insoluble in excess.

Alkalic carbonates produce a precipitate insoluble in excess. From dilute solutions it is crystalline. The precipitate is insoluble in ether; hence distinguished from quinine.

Gives no green colour with chlorine (or bromine) water and ammonia.

Gives no red colour with the above, and a few drops of potassium ferrocyanide previously added.

Concentrated  $\text{H}^2\text{SO}^4$ , heated with cinchonine, rapidly blackens; hence distinguished from quinine. The salts are soluble in water, and the solutions taste bitter; but the alkaloid being insoluble, has no taste; hence, mixed with sugar, it is sometimes advertised as sweet quinine.

#### SALICINE.

$\text{C}^{13}\text{H}^{18}\text{O}^7$ .

Although this substance is not an alkaloid, but rather a glucoside, it may be mentioned here as being sometimes used as an adulterant of quinine or its salts, or as a substitute, possessing a bitter taste and some febrifuge qualities.

Easily detected by giving a bright red colour, with strong sulphuric acid, especially on warming.

When distilled with dilute sulphuric acid and potassium chromate, the distillate has a peculiar smell, and gives a violet colour with ferric chloride (salicylous acid). When boiled with dilute sulphuric acid, the solution becomes turbid from formation of saliretine; and the liquid contains grape sugar (glucose), which may be detected as described hereafter; the character of all glucosides.

#### STRYCHNINE.

$\text{C}^{21}\text{H}^{22}\text{N}^2\text{O}^2$ .

Forms distinct prismatic or octahedral crystals; is scarcely soluble in  $\text{H}^2\text{O}$ , but communicates an intensely bitter taste; is soluble in common alcohol, chloroform, amylic alcohol and ether, especially when freshly precipitated.

When dissolved in  $\text{H}^2\text{SO}^4$ , and a small crystal of potassium bichromate added, a fine purple colour is produced, changing rapidly to red. The crystal should be moved through the  $\text{H}^2\text{SO}^4$ ; purple streaks are visible, with the most minute trace of the alkaloid.

Other oxidising substances may be used, such as  $\text{PbO}^2$ ,  $\text{MnO}^2$ ,  $\text{K}^3\text{Cfy}$ ,  $\text{KMnO}^4$ ; ceric oxide has been recommended.

Strychnine even in dilute solutions is precipitated as a yellow powder by potassium chromate, white by  $\text{KCys}$ , molybdo-phosphate and tungsto-phosphate of sodium. These precipitates can be used for the chromic test.



Pure strychnine is not coloured by  $\text{HNO}^3$ , but the commercial alkaloid usually contains brucine, which gives a red colour.

Strychnine is not altered by warming with  $\text{H}^2\text{SO}^4$ , a fact which is of great importance in freeing it from impurities.

The solution of KI with a few drops of  $\text{Fe}^2\text{Cl}^6$  gives a brown precipitate in very dilute liquids.

The alkaloid is precipitated by alkalies and their carbonates, and is insoluble in excess.

The action on a small frog immersed in the solution is also very characteristic.

#### SANTONINE.

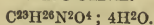


So many accidents, some of them fatal, have happened even in our comparatively small community, from mistakes arising from the administration of one of these principles for the other, that it has been thought advisable to introduce it here, although not an alkaloid.

#### *External Appearances.*

1. When in powder, they are not distinguishable.
2. When crystallised in large quantities, santonine appears in fine scales, strychnine in distinct prisms.
3. When from dilute solutions, the strychnine appears in acicular crystals, the santonine in plates.
4. When pure, they are both quite colourless.
5. When exposed to sunlight santonine turns yellow, strychnine does not; hence crystals of the latter may be readily detected in a mixture with santonine, such as unfortunately occurred some time since.
6. Strychnine dissolves readily in warm dilute acids—the solution has an intensely bitter taste; santonine does not—the liquid has little or no taste.
7. Strychnine (commercial) gives a reddish colour with  $\text{HNO}^3$ ; santonine does not.
8. Strychnine in the smallest particles gives fine evanescent purple colour with chromic acid (as above); santonine does not.

#### BRUCINE.

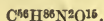


Does not often come under notice as a poison; it is almost always present in commercial strychnine, and causes the red colour produced therein by  $\text{HNO}^3$ .

With  $\text{HNO}_3$  gives a very bright red colour, not changed by reducing agents.

With chromic acid produces no purple, but only a pink, changing to yellow.

#### VERATRINE.



With  $\text{HNO}_3$  forms resinous lumps, which dissolve to a colourless solution.

With  $\text{H}^2\text{SO}^4$  forms similar lumps, which dissolve to a faintly yellowish liquid, changing to reddish yellow, and finally to deep blood red. The colour disappears after some hours.

#### CURARINE

Gives the same, but more permanent, colour with chromic acid, &c., as strychnine, but is soluble in water, reddened by  $\text{H}^2\text{SO}^4$ , and is not soluble in ether; hence easily distinguished from that alkaloid.

#### ACONITINE.

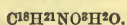


Is said to give a permanent pink colour with syrupy phosphoric acid.

With the  $\text{KI}$  and  $\text{Fe}^2\text{Cl}^6$  solution gives a brown precipitate.

Produces, when placed on the tongue, even in very minute quantity, a tingling and sensation of numbness.

#### CODEINE.



May sometimes act as a poison if taken in too large doses.

Is precipitated by alkalies, but insoluble in excess.

#### NARCOTINE.



Alkalies and their carbonates precipitate the alkaloid as a white powder, insoluble in excess of reagent, easily dissolved on shaking with ether.

It dissolves in  $\text{HNO}_3$ , and gives a yellow colour on heating.

It dissolves in  $\text{H}^2\text{SO}^4$ , with a faint yellow colour; the addition of a very minute trace of  $\text{HNO}_3$ , or a small crystal of  $\text{KNO}^3$ , produces a red colour.

## SEPARATION OF ALKALOIDS FROM ORGANIC MIXTURES.

Considerable difficulty attends this operation, as it is essential to prepare the alkaloid in a state of purity. It depends on the extraction of the organic mixture by some acid, the removal of impurities from the salt by ether, amylic alcohol, or other means, the separation of the alkaloid in a free state, and its removal from the solution by shaking with some appropriate solvent; further, the purification of the substance so obtained by a repetition of the same process or some other.

Only a brief outline will be here given. The student is referred to Presscott's Proximate Analysis, Otto's *Ausmittlung der Gifte*, and other works.

The organic substance, stomach, contents, viscera, &c., is digested for twenty minutes with alcohol, or water acidulated with a little tartaric, oxalic or hydrochloric acid; the whole filtered and well washed out, the filtrate evaporated to near dryness, and redissolved in water. By this, or a repetition of it, much impurity, such as fat, &c., is got rid of.

The acid solution is now shaken for some minutes with ether or amylic alcohol, in which salts of most alkaloids are insoluble, for the purpose of still further removing impurities, and the aqueous solution drawn off by a burette.

The acid solution is now rendered alkaline by ammonia or sodium bicarbonate, and immediately shaken with ether, chloroform or amylic alcohol. It is better to add the alkali and solvent at once, as the freshly precipitated alkaloids are more readily soluble than when they have become crystalline. The solution thus obtained being drawn off, is evaporated to dryness (volatile alkaloids cannot be rendered quite dry, but remain oily) in a water bath, and the operation, as above, repeated if necessary. In the case of strychnine, a good plan is to warm the residue with  $\text{H}^2\text{SO}^4$ , by which the alkaloid is not altered, but most organic matter charred, and the same operation repeated. The solution may then be evaporated to dryness in a capsule, or preferably allowed to drop gradually on to a heated glass slide or porcelain dish, whereby the substance is confined to one spot. Several of these slides or capsules should be prepared, unless the

body be immediately detected, and it be thought necessary to ascertain the quantity present; in such case the agitation with ether, &c., should be several times repeated.

The proper tests can then be applied to these residues. The alkaloids may also be precipitated from their purified solution by picric, phospho-molybdic, phospho-tungstic acid, mercuric-potassium iodide, cadmium-potassium iodide, and tannic acid, in presence of  $H^2SO^4$ , but not always in that of  $HCl$ .

The alkaloid itself can be extracted from these precipitates by drying with sodium carbonate and digestion with ether, chloroform or amylic alcohol. Strychnine is more easily soluble in the two latter than in ether.

## URINE.

The specific gravity of healthy urine varies considerably under certain circumstances; the average may be said to lie between 1015 and 1025. If under the first or over the latter (Bright's disease, diabetes), the urine may be considered abnormal. The S.G. may be taken in the usual way by the urinometer or bottle; but a more accurate method, applicable to almost all liquids, is to attach a glass stopper to a piece of thin platinum wire or silk, and ascertain its weight in air and its loss of weight in water at  $15^{\circ}C = A$ . These weights may for convenience be marked on a moveable label attached to the wire. Now ascertain the loss of weight in the urine at  $15^{\circ}C = B$ ; the S. G.  $= \frac{B}{A}$ . It should be remarked, that in ascertaining the S.G. of urine, the whole passed during the day should be mixed and then experimented on.

Urine is usually slightly acid, nearly neutral after meals, becomes rapidly alkaline on keeping, from formation of ammonium carbonate from the urea; this change sometimes takes place in the bladder, so that the fresh urine is strongly alkaline, and may even effervesce with acids. This is especially the case with diseased urine containing mucus; the ropy sediment which forms on standing often contains large crystals of the triple phosphate. See plate of triple phosphate.

Healthy urine is quite clear, and forms no distinct deposit on standing over night. If a sediment is formed which vanishes on addition of the morning's urine, or on warming, it is urate of ammonium, or more frequently of sodium, rarely of calcium; if the sedi-



ment does not disappear on warming, but on the addition of acetic acid, earthy phosphate; if it remains undissolved, probably calcium oxalate. This latter is not unfrequently met with, and is best recognised under the microscope.

#### UREA.



Urea exists in the urine in variable quantities, one and a-half per cent. being about the average, and constituting about one-third of the solid contents. The quantitative determination is attended with considerable difficulty; but its presence and amount may be approximately ascertained by evaporating a measured bulk to one quarter, cooling, mixing with equal amount of  $\text{HNO}^3$ , and allowing to stand. The nitrate will crystallise out. For crystalline forms see plates.

It passes rapidly when in the urine into ammonium carbonate —  $\text{COH}^4\text{N}^2 + \text{H}^4\text{O}^2 = \text{N}^2\text{H}^6\text{CO}^3$ . On the other hand, ammonium carbonate can be converted into urea by heating to  $135^\circ$   $\text{CN}^2\text{H}^6\text{CO}^3 - \text{H}^4\text{O}^2 = \text{CN}^2\text{H}^4\text{O}$ . The carbonate is unstable and evolves  $\text{NH}^3$ ; hence the pungent smell of decaying urine.

#### URIC ACID.

Uric acid can be detected in the urine by adding some  $\text{HCl}$ , and allowing to stand for twenty-four hours or more, a reddish sediment is formed which can be tested for uric acid in the usual way. The quantity present in human urine is small, about one part in 1000, or 7-8 grains in twenty-four hours. The urates may be examined as described under Sediments.

#### HIPPURIC ACID.

As the importance of this substance, although always present in the urine, and sometimes in notable quantity, has not yet been ascertained, it may be sufficient to merely mention its occurrence.

#### SALTS.

The chlorides, sulphates and phosphates of alkalis and earths can be detected in the usual manner, either in the fresh urine, or after evaporation and incineration. They amount to about fifteen parts in 1000.

#### FAT AND CHYLE.

The fat and chyle corpuscles can be recognised under the microscope, the former soluble in ether, the latter not so. The fat globules resemble those of milk, but seem to be more readily attacked by ether. Kiestein, which forms a glistening pellicle on some urine

(formerly considered a proof of pregnancy), is apparently some organic matter with entangled crystals of earthy phosphates.

#### EPITHELIUM CELLS.

These, as well as renal casts, sarcinæ, spermatozoa, and to a certain extent blood corpuscles, are objects of microscopical rather than chemical investigation.

#### ALBUMEN

Is best detected by  $\text{HNO}^3$ , or by heating. It has been proposed to apply the  $\text{HNO}^3$  test by pouring the urine on to the surface of the acid, and noticing whether a film is formed at the point of junction; the process is said to be open to some fallacies.

In applying the method by heat, the boiled urine should be compared with some in its natural state; or a test tube may be nearly filled with urine, and the upper half after boiling compared with the lower, to which heat has not been applied. In either case a drop of  $\text{HNO}^3$  should be added, as the turbidity may arise from earthy salts, which of course will disappear. The urine may originally have been turbid, in which case it is well to warm gently to dissolve urates, or make slightly acid with acetic, not nitric acid. If the urine be alkaline, no precipitate will be formed on boiling; it must be acidified with Ac.

A very slight turbidity may arise from presence of pus, or blood; in the latter case the urine will be more or less coloured, and will deposit a sediment on standing, in which the red corpuscles may be seen. It may be necessary to filter a turbid urine, if the opacity is not removed by heat or Ac.

For detecting albuminous bodies other than albumen, a solution of mercuric nitrate is recommended, which gives a red colour, especially on warming.

#### GRAPE SUGAR

Is said to be always present in healthy urine to the extent of about 0.1 per cent., and in old urine it may result from the decomposition of the glucoside indican, which yields sugar and indigo. In diabetes the quantity is enormously increased, varies according to diet, &c., and sometimes disappears entirely towards the termination of the disease. For quantitative determination, see Fresenius, or other works.

Trommer's test, depending on the fact that grape sugar in contact with potassa or soda and cupric oxide forms a clear blue solution,

which on warming or standing produces insoluble red cuprous oxide, is perhaps the best, with certain precautions. A test tube being half filled with the urine, about half as much solution of pure potassa or soda is added, and then a few drops of a weak solution  $\text{CuSO}^4$ . Care must be taken not to add too much, as otherwise a permanent precipitate will be formed. It is better to use the reagents in the above order. A precipitate is produced, which dissolves on shaking. If by accident too much  $\text{CuSO}^4$  has been used, some more urine must be added. On gently warming, an orange or red precipitate will be formed (also on standing twenty-four hours); on boiling, this turns brown, hence it is better not to heat so far. It has been recommended to use Fehling's solution, *i. e.*, potassium tartrate with potassa, and  $\text{CuSO}^4$ ; but as the reagent is apt to decompose, it must always be examined by boiling before each experiment to see that no red precipitate is formed before the addition of the sugar. It does not appear to possess any great advantage over the above test if carefully applied. If it should be used, a large excess compared to the urine must be employed, unless the quantity of sugar be exceedingly small.

Urine containing grape sugar when boiled with potassium hydrate, becomes dark coloured. If albumen be present, it must be previously removed by boiling.

The fermentation test, production of  $\text{CO}^2$ , may also be applied by inverting a tube half filled with urine over mercury, or a full tube over urine, throwing up into it some yeast and placing in a warm situation. Gas collects in the top of the tube, but the operation takes some time.

#### URINARY CALCULI.

These are either destructible by heat or not so; but almost all calculi contain traces of mineral matter, and the earthy ones generally blacken more or less on the application of heat. The calculi are often complex, consisting frequently of a kernel of ammonium urate, with coating of earthy phosphate. The mulberry stone, calcium oxalate, often accompanies phosphates. The different layers can easily be separated and examined by same tests as applied to the sediments. Those that are destroyed by heat, leaving only a small residue, are uric acid, ammonium urate, cystin and xanthin—both these latter very rare; of xanthin, the writer has not been able to obtain a specimen.



Some phosphatic calculi, especially those found in the kidney, are scarcely soluble in acetic acid, they must be dissolved in HCl, and reprecipitated. The mulberry calculus is insoluble in acetic acid, but soluble with effervescence after heating; soluble in HCl before heating, reprecipitated by  $\text{NH}^3$ , or sodium acetate.

Other calculi are so rare that they need not be here mentioned; the prostatic calculi are usually phosphates.

#### CYSTIN.

This substance forms a soft friable calculus, or appears sometimes as a crystalline sediment. The calculus or sediment may be dissolved in ammonia, and a drop allowed to evaporate on a slide, the very distinct hexagonal form will be seen, as in plate (cystin). The calculus generally contains some few per cent. of earthy phosphates, which are left behind on treating with alkali. The cystin, when heated, evolves a peculiar smell, and the urine itself has sometimes a very disagreeable odour.

Liebig's test, depending on the presence of sulphur, consists in adding KHO to a solution of lead acetate till the PbO is redissolved, and then boiling with some of the alkaline solution of cystin, when black PbS is produced. The writer found, on making some experiments with a fine sample of this very rare calculus, lately presented to him by Dr. Zimmerman, that the reaction takes place more readily by adding the solution of cystin in  $\text{NH}^3$  to lead acetate, to which a few drops of  $\text{NH}^3$  have been added.

It has been stated that Liebig's test is fallible, as other substances, such as albumen, which contain sulphur, would give the same reaction. The writer has found that by employing  $\text{NH}^3$  instead of KHO, this difficulty is got over. Albumen added to urine, and treated as above, does not cause any blackening.

#### URINARY DEPOSITS.

The sediment is either soluble or insoluble in the warmed urine; in the former case it is ammonium, sodium or (?) calcium urate; in the latter, uric or hippuric acid, cystin, phosphate or oxalate.

The ammonium urate may be tested for uric acid by  $\text{HNO}^3$  (see Uric Acid), and for  $\text{NH}^3$  by the ordinary HKO test, or perhaps more accurately by placing over the capsule in which the experiment is made a watch glass moistened with water; after a time, Nessler's test may be applied to the solution of  $\text{NH}^3$  obtained.



Sodium and calcium can be detected by the usual tests.

The insoluble sediment, when treated with acetic acid, yields up the earthy phosphates, leaving uric acid, hippuric acid and calcium oxalate, the latter soluble in HCl, the hippuric acid in alcohol, from which the distinct crystals can be obtained. A portion of the moist sediment may be placed on a slide covered with thin plate, examined under microscope as to crystalline form, and a drop of acetic acid placed in contact with the edge of the plate. The solubility, insolubility, or change of form, give means of recognition.

For cystin, see under Urinary Calculi.

The earthy phosphates, dissolved in acetic acid, can be reprecipitated by  $\text{NH}_3$ , and the precipitate examined in the usual way employed for insoluble salts. The reactions of water, acetic and hydrochloric acids, are, as Attfield remarks, generally sufficient, as the deposits are seldom complex.

#### BILE.

1. When in a tolerably pure state, may be detected by adding to the solution three or four grains of cane sugar, and then sulphuric acid until the whole becomes hot. A beautiful purple colour will be produced.

2. The liquid containing the sugar, with a few drops of acid, may be evaporated over a water bath, when the same colour will appear.

3. A more delicate way is to dip a strip of filtering paper into the solution, allow it to dry, and pour on a drop of sulphuric acid, allowing most of it to run off; fine violet colour is produced.

4. A strip of paper dipped into a solution of bile and dried, will give a violet colour, changing to yellow, or brown with a drop of nitric acid.

5. The solution of bile may be precipitated with basic lead acetate, the precipitate washed, mixed with sodium carbonate, dried and extracted with absolute alcohol, filtered, the filtrate evaporated, and the residue dissolved in a very small quantity of water. One drop of this mixed with one drop of dilute sulphuric acid (4 : 1), with a trace of sugar, and evaporated, will give the above colour.

For testing urine for bile, it should be evaporated to syrup, extracted with absolute alcohol to remove albumen and salts, the alcoholic solution evaporated, residue dissolved in water, precipitated with lead salt, and precipitate treated as above.

Biliary calculi, or gall stones, consist principally of cholesterine with more or less colouring matter. By boiling ether, the cholesterine may be extracted, and will separate out in crystalline scales on cooling or evaporation.

The bile pigments may be detected by allowing a drop of urine and  $\text{HNO}^3$  to mix gradually on a white porcelain plate, a red colour will appear at the line of junction with every urine, but in presence of the bile, a play of colour from green to violet, blue and red.

#### MUCUS AND PUS.

Mucus is often found in morbid urine in large quantities ; it forms a viscid sediment, which when shaken up, renders the urine ropy. The pus globules are readily suspended and recognisable under the microscope. (See Plate). Mucus is coagulated by acetic acid, which dissolves the crystals of ammonium-magnesium phosphate which are often entangled in it and are frequently of large size, of the form given in Plate. Urine containing mucus is generally alkaline, from the decomposition of the urea into ammonium carbonate, but is not coagulated by boiling or by nitric acid. If the urine contains pus, a slight turbidity may be observed, from the presence of a trace of albumen in the pus. The opacity of the pus globules when treated with acid, as seen under the microscope, is probably owing to this cause.

Pus appears as globules about  $\frac{1}{2000}$ th of an inch in diameter, often mixed with ropy mucus. With a power of 400 they appear granular, more especially on addition of acetic acid. With potassium hydrate they become jelly-like, hence distinguished from mucus.

#### COLOURING MATTER.

Numerous colouring matters have been extracted from urine ;\* the most common is the red, erythric acid, produced by the partial oxidation of uric acid. The dark colour of urine in cases of fever seems to be due to the formation of this substance—erythric acid of Brugnatelli, murexide of Liebig. Sometimes the red colour arises from the presence of blood, recognisable by the usual tests. Sometimes the urine is blue, from the presence of indigo, formed by the oxidation of indican, which has been shown to exist in urine in small quantities.† Bile may also produce a yellowish colour.

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\* Thudicum.

† Schunck.

## BLOOD AND BLOOD STAINS.

The chemist is often called upon to determine whether stains on cloth, wood or metal, have been caused by blood or other substances, usually of vegetable origin. A blade of steel on which a clot of blood has settled, will generally, when gently heated, allow it to be peeled off. A fruit stain will not so act, the stain chars.

On examination of any substance which may be supposed covered with blood, the following may be given as the mode of analysis :

1. Soak the substance in water or dilute glycerine.
2. A reddish solution is obtained, not altered to any great extent by addition of ammonia. Most vegetable colours are turned blue.
3. A portion of the dry substance, heated in a tube, with a piece of turmeric (or red litmus) paper in the mouth, causes a brown (or blue) colour. Vegetable matters as a rule give off *acid* vapours, turning blue litmus red.
4. A drop of the red solution, obtained as above, is mixed with a drop or two of tincture of guaiacum ; to that is added a drop of hydroxyl solution. Old oil of turpentine is the handiest article for this purpose ; it almost always contains sufficient peroxide of hydrogen for the experiment. A blue colour will be produced. A green may result from the guaiacum alone, but not at all to be confounded with the blue from blood. The cause of this colour is not known to the writer.

A blood stain may be moistened, allowed to stand for some time, and a spot produced on filtering paper by pressure. This can then be treated with guaiacum and hydroxyl (? ozone) as above ; the most minute trace can in this way be detected.

Nitric acid produces a precipitate in the above red solution owing to presence of albumen. If the quantity be very small, it is better to place some nitric acid in a short test tube, and pour the liquid carefully on to the top of it so as not to mix ; an opaque film at the point of junction will indicate albumen. This method is especially valuable in examining urine, but objections have been raised to it.

Mercuric chloride will also produce a precipitate. Picric acid may also be employed.

If chlorine be passed through the red solution, a flocculent precipitate and greenish colour will be produced. The liquid portion,



filtered off and boiled till all chlorine is expelled, will give a faint red colour with potassium sulphocyanate, best seen by looking down into the tube, held over a white surface, from presence of iron.

### ADULTERATIONS.

Any substance of inferior value, mixed with a saleable article of greater value, must be considered as an adulteration. Any person vending an article marked pure or genuine, which contains such inferior substance, must be considered as guilty of adulteration. If the body sold is not so marked, then the consumer must run his own risk. The adulteration may be mechanically injurious, as for instance in the case of pigments, or directly so in a medicinal point of view, as in the case of drugs. Only a few instances will be mentioned in the following pages, as examples for practice in this very extensive and important subject.

### PRACTICE EXAMPLES.

#### POTASSIUM IODIDE

Often contains iodate, which gives a yellow or brown colour to the otherwise colourless salt. Its presence may be detected by adding tartaric acid to the solution; a brown colour, caused by iodine, will be produced, the iodine detected as described under Hydriodic Acid. Potassium carbonate may be detected by its being insoluble in boiling alcohol, by effervescence on addition of dilute  $\text{HNO}_3$  to solution, by giving a precipitate with  $\text{CaCl}_2$ . The salt may also be adulterated with chloride or bromide, the latter compound being at present so much cheaper than the iodide. A portion of the salt should be dissolved in water, fully precipitated by  $\text{AgNO}_3$ , the precipitate washed and treated with  $\text{NH}_3$ , all the chloride (and a portion of bromide) will be dissolved, and can be reprecipitated by addition of  $\text{HNO}_3$ . On warming the residue with strong  $\text{NH}_3$ , the bromide can be dissolved out, leaving the iodide. The solution can be treated with  $\text{HNO}_3$  and the precipitated bromide examined by appropriate tests. The chloro-chromic test (see Chromium) for chloride is not well applicable if bromide be present unless by passing the vapours into dilute  $\text{NH}_3$ , when a yellow colour will indicate the presence of chromic acid, detected also by usual tests. Iodine and bromine do



not form compounds analogous to chlorochromic acid, perhaps more correctly called oxychromic chloride,  $\text{CrO}^{\circ}\text{Cl}^{\circ}$ .

#### POTASSIUM BROMIDE

Sometimes contains bromate, detected as iodate; the adulteration by chloride may be detected as above described.

#### MERCURIC OXIDE

Is occasionally adulterated with red lead, or if in fine powder, with red brickdust. The latter is easily detected by being insoluble in hot dilute  $\text{HNO}^3$ , the former by remaining as a yellow powder on strongly heating in a capsule.

#### MERCUROUS CHLORIDE

Sometimes contains mercuric chloride, easily detected by boiling for a few minutes with water, filtering and applying to the solution the appropriate tests for mercuric salts.

#### MERCURIC SULPHIDE

May contain the same impurities as the oxide. The sulphide is volatile at a high temperature, the above named substances are not.

#### WHITE LEAD.

This substance is a mixture of lead carbonate and hydrate in somewhat variable proportions. Its use as a pigment depends on its freedom from colour, its *covering* power, and its tendency to gradually unite (as oxide) with the fatty acids contained in the oil employed. Lead oxide, under the name of *dryers*, is often used to accelerate this change. Barium sulphate being of nearly the same gravity, is often used as an adulterant, but being, more especially in its native form, highly crystalline, does not possess the *body* or covering power, and does not act on the oil. Calcium carbonate and sulphate are sometimes, but rarely employed, owing to their low gravities.

Zinc oxide being of a very pure white, is sometimes used to improve the colour, and is not so objectionable an adulteration as the above.

The white lead is easily soluble in hot dilute  $\text{HNO}^3$ , the residue being  $\text{BaSO}^4$ . From the very dilute solution the lead may be precipitated by  $\text{H}^2\text{SO}^4$  or  $\text{H}^2\text{S}$ , the filtrate neutralised by  $\text{NH}^3$ , and the

zinc precipitated by  $\text{Am}^2\text{S}$ . In the filtrate from this the calcium may be detected by appropriate tests.

Very few specimens of commercial white lead are really pure; out of sixteen specimens examined by the writer some time since, only one was found absolutely pure, although in some cases the samples were marked *genuine*. Some samples contained forty-eight per cent. of  $\text{BaSO}_4$ . The method of determining the quantities lies beyond the scope of this small work.

#### PARIS GREEN—COPPER ARSENITE.

This pigment being now so much used as a means of destroying the Colorado potato beetle, it has naturally been adulterated, and generally with the above mentioned  $\text{BaSO}_4$ . This can be detected by treating the pigment with  $\text{NH}_3$  or dilute  $\text{HNO}_3$ , which dissolves the arsenite. My friend, Dr. Ellis, has found as much as twenty per cent. of  $\text{BaSO}_4$  in some samples, and also Prussian blue added in small quantity to deepen the colour, lowered by the use of the white  $\text{BaSO}_4$ . Salts of calcium are also sometimes used; these can be detected in the solution by precipitating the arsenic and copper by  $\text{H}^2\text{S}$ , and testing the filtrate for lime salts.

#### CHLOROFORM

Is sometimes mixed with alcohol, in which case the specific gravity will be less than 1.49, which it should have. The impurity can be detected by boiling with potassium chromate and sulphuric acid, when a green colour will be produced; also by the iodoform test. Both ether and alcohol can be estimated by shaking a measured quantity of chloroform with water in a burette; if pure, the bulk will not be diminished. The drug is sometimes impure from the presence of acids, recognised by test paper; methyl compounds are turned brown by warming with sulphuric acid. Chloral hydrate is said to become acid from formation of hydrochloric acid. In a very acid sample examined by the writer this acid was not present in a free state, but some other, probably formic or acetic.

#### QUININE, OR ITS SALTS.

Earthy matters, such as chalk, gypsum, &c., can be readily detected by burning the substance on platinum foil. It must be remarked, however, that long incineration is required to burn off all the carbon.

Cinchonine may be detected by precipitating the solution in dilute acid, by sodium carbonate and shaking with ether, quinine dissolves, cinchonine does not.

Starch, by boiling with dilute sulphuric acid, and testing for sugar, or by the iodine test. But in this case it would be better to extract the quinine first of all by maceration in dilute acid, to avoid the brown colour produced by iodine solution.

Sugar, by boiling with acid, and usual test.

Salicine, by  $\text{H}^2\text{SO}^4$ . See under Salicine.

Cinchonine may be detected by making a solution of the salt in dilute  $\text{H}^2\text{SO}^4$ , adding a slight excess of  $\text{Na}^2\text{CO}^3$  and shaking with ether; on allowing to stand, a film will be seen at the point of junction of the two liquids. Also by adding to the above solution on a microscope slide a drop of very dilute solution (6 grs. in  $\frac{1}{2}$  oz.  $\text{H}^2\text{O}$ ) of  $\text{KCy}$ , and putting on a thin cover. The quinine salt will form long slender crystals, that of cinchonine well formed prisms.

#### MILK.

Although much has been said and written about the adulteration of milk, from several thousands of analyses made during the past five years, both in Europe and on this side of the Atlantic, the writer has met with but one decided case, unless the admixture of water or the abstraction of cream can be considered as such.

Ordinary milk should contain about eleven or twelve per cent. of solid ingredients; anything under that would show either poor or underfed cows, or wilful adulteration. It is curious to remark that the milk from cows in Lower Canada is generally much richer than in Ontario, the average being fifteen, sometimes seventeen, per cent. of solid matter.

The analysis of milk being essentially quantitative, cannot well find a place in this small work. The reader is referred to Wanklyn, who evaporates 5 CC to dryness; or Baumhauer, who preferably uses pure sand for assisting the process. The increase of weight gives the solid contents; the diminution in weight after washing with ether, gives the butter or fat; the further diminution on washing with water, gives the sugar, which may be more accurately determined by Fehling's solution; but it must be remembered that milk sugar does not reduce  $\text{CuSO}^4$  readily unless previously converted into grape

sugar or galactose by boiling some time with acid. The amount of ash left on incinerating a certain quantity also affords some criterion as to the purity of the milk. It is said that salt is sometimes added to raise the specific gravity of milk lowered by addition of water. The estimation of the NaCl will at once show this adulteration.

#### BEER.

The most common adulteration is salt, chloride of sodium ; of this fifty grains is allowed to the gallon ; anything over that may be condemned. It must be borne in mind that NaCl is almost always present in minute quantities in vegetable infusions.

Picrotoxine, black extract, cocculus indicus, is sometimes used for increasing the intoxicating power of beer. There is no very good test for this substance, but it may be extracted by the ordinary plan for alkaloids.

Picric acid is said to have been used for increasing bitterness of beer. Detected by boiling strand of woollen fibre, which acquires a yellow colour.

#### WINES.

Some wines are manufactured and colour given to them by aniline compounds. A piece of gun cotton placed in pure wine acquires no colour, in spurious it becomes tinged. Pure wine shaken with ether gives no colour thereto; wine coloured by aniline compounds gives a tinge to the ether, removed by addition of HKO, and reproduced by exposure to air. The amount of alcohol determined as already described. The artificial colouring matter, it is said, may be detected by a cube of gelatin; in pure wine the colour enters in scarcely one quarter inch, in spuriously coloured the whole becomes tinged.

#### VINEGAR

May contain  $H^2SO^4$ , HCl, &c.; detected by usual tests. Also iron from error in construction of plant; detected by common tests.

#### SPIRITS.

It is well known that most of these are made from alcohol, called high wines, with some colouring matter (burnt sugar), and some trace of flavouring essence. The detection of fusel oil, amylic alcohol, in some kinds (Bourbon) of whiskey has been already alluded to.



Whether the very small quantities of flavouring essences, principally compound ethers, may be injurious or not, is a question. A writer on vinegar, in order to test the question, devoured one pound of pear drops, flavoured of course by amylic acetate; the only result was that he did not care for eating any more pear drops.

Butyric ether is used for pine apple flavour. Pelargonic ether is said to exist in the quince; something like sebacic ether in the apple or melon. Probably the flavours of fruits are owing to the presence of such compounds, and yet we eat them without fatal results.

#### CONFECTIONERY.

Usual adulteration is starch; easily recognised under the microscope and by its insolubility in cold water, and by iodine test. Not unfrequently salts of copper, and even arsenic, are used in imparting colour to the confection itself, or to the paper in which it may be inclosed.

#### TESTS FOR SOME OF THE MORE COMMONLY OCCURRING MINERALS.

**SHELL MARL.**—By washing with water and pouring off the lighter particles, fragments of shells may readily be detected. The mineral dissolves easily in dilute nitric acid, gives a precipitate with oxalic acid and ammonia; the precipitate, separated after warming, by filtration, and the filtrate tested for magnesia by means of sodium phosphate. The nitric solution heated with ammonium molybdate may give (rarely) a faint yellow precipitate or colour, owing to presence of phosphate.

**GYPSUM**, calcium sulphate. See under Examination of Insoluble Substances. Some samples from Caledonia are absolutely pure  $\text{CaSO}_4 + 2\text{H}_2\text{O}$ , perhaps from particle of bone.

**HEAVY SPAR**—barium sulphate—may be examined in the same way.

**LIMESTONES** may be examined in the same manner; many contain magnesia (Rockwood). They sometimes contain bituminous matter, which causes them to evolve a disagreeable smell when pounded or heated. Stinkstone; Collingwood shale.

**CALCSPAR** is generally met with in the rhombic or the scalenohedral form ; effervesces even with weak acids ; can be scratched with a knife.

**FLUORSPAR** is usually crystallised in cubes ; treated with sulphuric acid evolves a gas which corrodes glass ; does not effervesce with weak acids.

**APATITE**—calcium phosphate, with calcium fluoride and chloride—is usually of a greenish or reddish colour ; occurs generally in six-sided prisms ; a gigantic crystal may be seen in the School of Practical Science ; when in powder dissolves readily in nitric acid. The phosphoric acid can be detected by heating the solution with ammonium molybdate, when a yellow precipitate is formed ; or by adding silver nitrate to a solution obtained by boiling an excess of the mineral with a very little nitric acid, a yellow precipitate will be formed. A considerable excess of the AmMo must be added, otherwise only a very indistinct reaction will be produced. The addition of ammonia dissolves the silver phosphate, but of course precipitates the original salt. The lime can be detected by adding oxalic acid and excess of sodium acetate to the acid solution. A light coloured pyroxene is often mistaken for apatite, readily distinguished by being much harder, and quite insoluble in nitric or hydrochloric acid.

**HÆMATITE**—ferric oxide—is not magnetic ; when in fine powder dissolves in hydrochloric acid. The addition of nitric acid to the hot solution does not change its colour. The iron can be detected by usual tests ; the ore gives a brown red streak on rough porcelain.

**MAGNETIC IRON ORE.**—Attracted by the magnet, the hot hydrochloric solution turns first black on the addition of nitric acid, and then brownish yellow. Ore gives a black streak.

**OCHRE**—hydrated ferric oxide, more or less pure—is of a yellow colour, soft and easily soluble in acids. The iron can be detected by usual tests.

**TITANIC IRON ORE.**—Scarcely attracted by the magnet ; dissolves in hydrochloric acid only when very finely pounded. The titanic acid is best detected by fusing with an excess of potassium bisulphate ; dissolving in a large quantity of cold water and boiling for

a long time, a white or yellowish precipitate is formed. Finely pounded and dissolved in warm HCl, boiled with tin granulated, gives violet colour. Found at Chateau Richer and other localities on the Lower St. Lawrence, sometimes with magnetic sand.

**SULPHIDES**, with the exception of cinnabar (mercuric sulphide, not occurring in Ontario), are acted on by nitric acid, red fumes are evolved, and the solution contains sulphuric acid. In the case of galena (lead sulphide), the sulphuric acid remains combined with the lead oxide, as insoluble lead sulphate. (See Galena.) The sulphur can generally be detected by heating a portion of the ore in a tube, open at both ends, held slanting, and testing the gas evolved by iodic starch paper. Some sulphides, such as those of zinc, antimony and bismuth, may be dissolved in hydrochloric, with evolution of hydrosulphuric acid, and the solutions examined by the proper tests. The sulphides of antimony and bismuth have been found mixed with galena. See tests for these two metals.

**IRON PYRITES**, or ferric sulphide (Mundig), when heated strongly before the blowpipe, loses sulphur and forms a globule of ferrous sulphide, which is attracted by the magnet. The powdered ore is readily dissolved by nitric acid, which is not the case with gold, for which this ore is often mistaken. Gold will dissolve in aqua regia; while so-called golden mica, often mistaken for the metal, will not.

**COPPER PYRITES**, when gently roasted, moistened with hydrochloric acid, and exposed to the outer flame of the blowpipe, will impart to it a blue or green colour. The ore may be dissolved in nitric acid, the solution treated with excess of ammonia. A brown precipitate of ferric oxide will be formed, and a blue solution obtained.

**GALENA**—lead sulphide—when heated strongly on charcoal, will give a soft malleable bead of metallic lead, which may be dissolved in hot dilute nitric acid. The proper tests can then be applied to this solution. When finely powdered can be dissolved in hydrochloric acid.

**ZINC BLENDE**—sulphide—is soluble in the same acid. See tests for zinc.



**CHROMIC IRON ORE**, when fused with sodium carbonate and nitrate, or nitre, gives a yellow mass in which chromic acid may be detected. The ore must be very finely powdered. The formula is similar to that of magnetic iron ore, viz.,  $\text{Cr}^2\text{O}^3\text{FeO}$ . Occurs in the Lake Superior district, and in small quantities in many Canadian rocks.

**GRAPHITE**, when roasted, slowly burns away, leaving generally a small earthy residue, insoluble in ammonia.

**MOLYBDENITE**, when gently roasted, especially with sand, gradually loses its dark colour, leaving a yellowish residue, which dissolves partly in ammonia. The molybdic acid can be detected by sodium phosphate (see Phosphoric Acid), or by blue colour produced by zinc and hydrochloric acid. Occurs in Lake Superior district and in some Eastern Townships. Formula,  $\text{MoS}^2$ . The mineral might easily be mistaken for graphite; the streak is somewhat lighter.

**COPPER NICKEL**.—Arsenide of nickel, of a red or copper colour, when heated in a tube open at both ends, and held in a slanting position, produces a white crystalline sublimate of arsenious acid. The residue, or the mineral itself, dissolved in nitric acid by the aid of heat, diluted, filtered and treated with hydrosulphuric acid, yields a yellow precipitate of arsenic sulphide; the filtrate is green, and gives the usual reactions of nickel. In some samples from mines on Lake Superior the ore is mixed with metallic silver, sometimes in large quantities. The nitric solution will then give a precipitate with hydrochloric acid. Formula,  $\text{NiAs}$ .

**MISPICKEL, ARSENIDE AND SULPHIDE OF IRON**.—The ore is of a silvery white colour. When heated in a close tube gives a dark brown, or metallic ring of arsenic; if heated in an open tube gives a sublimate of arsenious acid. Dissolves in nitric acid. The solution may be freed from arsenic by means of hydrosulphuric acid, and the filtrate tested for iron in the usual manner. The sulphur detected in the nitric solution by the ordinary test for sulphuric acid. The ore often contains traces of gold.

**SILICIC ACID**—quartz—can be taken up by fusing sodium carbonate with effervescence; the fused mass dissolved in water, the addition of hydrochloric acid will cause a gelatinous precipitate, or the acid solution may be evaporated to dryness, and washed with



water; the silicic acid remains undissolved. Very pure quartz, with scarcely a trace of iron, can be found in veins traversing the rocks about Stony Lake; if heated red hot and thrown into water, it becomes brittle and can be readily powdered; if required quite pure, any trace of iron can be removed by hydrochloric acid. This is useful in many chemical operations.

The above examples may serve as practice for the beginner. For plans for discriminating all the ordinarily occurring minerals, consult Chapman's "Minerals of Canada."

#### ABSOLUTE ALCOHOL IN 100 PARTS.

S.G.		S.G.	
0.9928	— 4.00	0.9854	— 8.87
0.9921	— 4.40	0.9849	— 9.28
.9915	— 4.81	0.9843	— 9.69
9909	— 5.21	0.9838	— 10.10
9902	— 5.62	0.9832	— 10.51
9896	— 6.02	0.9827	— 10.92
9890	— 6.43	0.9811	— 12.15
9884	— 6.83	0.9800	— 13.00
9878	— 7.24	0.9790	— 13.80
9872	— 7.64	0.9785	— 14.63
9866	— 8.05	0.9775	— 15.04
9860	— 8.46	0.9760	— 16.28

The above numbers are taken from Brix' table, Bolley's Technology, page 221. Only such as may usually be required are introduced; for lower or higher percentages, refer to Fownes or Bolley. Convert percentage of absolute alcohol into proof spirit by multiplying by two nearly (2.04).

## ERRATA AND ADDENDA.

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PAGE 4.—The more recent and accurate numbers have not been introduced, such as “O = 15·96, Cl = 35·37, Ca = 39·9,” &c., &c.

“ 6.—Six lines from top, read “ate” for “ide;” ten lines from below, read “Na<sup>2</sup>” for “Na.”

	SPECIFIC GRAVITY.	GOLD PER CENT.	CARATS.
“ 43.—	17·84	91·67	22
	16·60	83·34	20
	15·42	75·00	18
	14·48	66·67	16
	13·65	58·33	14
	12·91	50·00	12

“ 43.—Nineteen lines from top, read “O<sup>3</sup>” for “O.”

“ 45.—Fifteen lines from bottom, read “S<sup>2</sup>” for “S.”

“ 48.—Six lines from top, read “NH<sup>3</sup>” for “NH.”

“ 53.—Four lines from top, read “K” for “H.”

“ 63.—Thirteen lines from bottom, read “formula Pb.<sup>3</sup>P<sup>2</sup>O<sup>3</sup>” after “ppte.”

“ 63.—Paragraph three: “Ammonium molybdate gives similar reaction with silicic and arsenic acids, easily distinguished from phosphoric.”

“ 64.—Nine lines from top, symbol for “Boron” is “Bo.”

“ 65.—Expunge entry “Arsenic Acid,” for which see p. 45.

“ 67.—Ten lines from top, add, “by action of NHO<sup>3</sup>.”

“ 81.—Eight lines from top: “It may be safer to heat this ppte., and calculate As<sup>2</sup>O<sup>3</sup> as from magnesium pyro-arsenate, Mg<sup>2</sup>As<sup>2</sup>O<sup>7</sup> 63·87 per cent.”

“ 84.—Eight lines from top: “The Na<sup>2</sup>CO<sup>3</sup> must be quite dry.”

“ 86.—Under HCy; “A watch glass moistened with Am<sup>3</sup>S or AgNO<sup>3</sup> may be suspended over the suspected liquid, covered by a jar; tests then as described.”

“ 98.—Sixteen lines from top, expunge first “C.”

“ 103.—Three lines from bottom, read “change” for “oxidation.”

“ 110.—Ten lines from bottom, add, “probably from trace of bone of some bird or animal. Shells seldom contain phosphate.”

“ 110.—Seven lines from bottom, correct as above.

“ 111.—Three lines from top, add, “Does not scratch a *copper* coin;” and six lines from top, add, “Scratches a *copper* coin.”



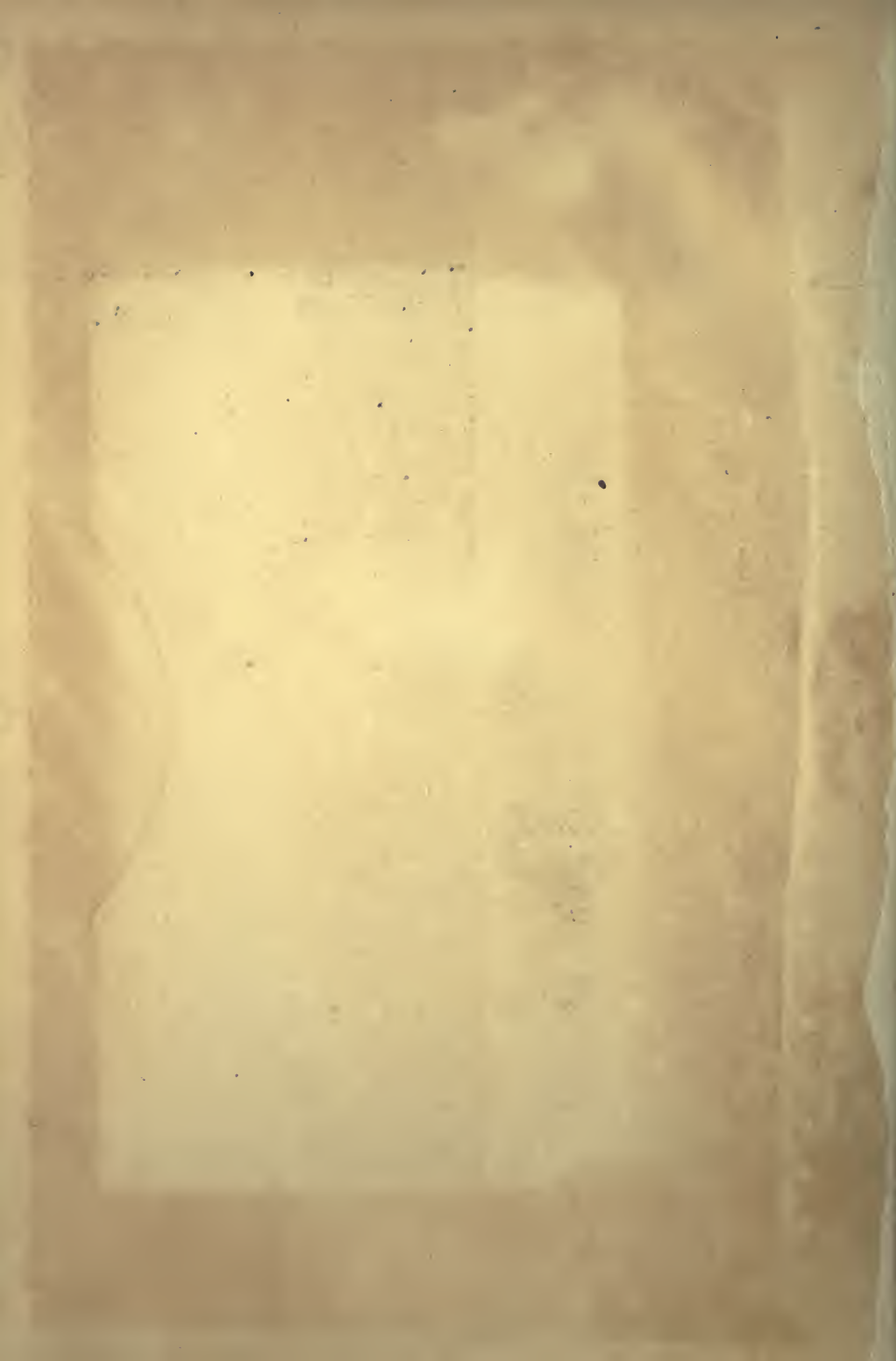
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Author Croft, Henry Holmes

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